

GLASS BONDED CERAMIC BODIES

By
K. N. RAO

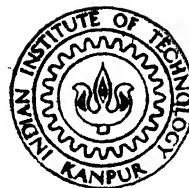
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DEPARTMENT OF METALLURGICAL ENGINEERING
INDIAN INSTITUTE OF TECHNOLOGY, KANPUR
APRIL, 1985

GLASS BONDED CERAMIC BODIES

A Thesis Submitted
In Partial Fulfilment of the Requirements
for the Degree of
MASTER OF TECHNOLOGY

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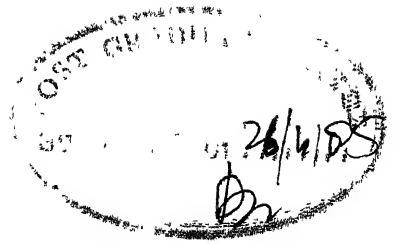
By
K. N. RAO

to the
DEPARTMENT OF METALLURGICAL ENGINEERING
INDIAN INSTITUTE OF TECHNOLOGY, KANPUR
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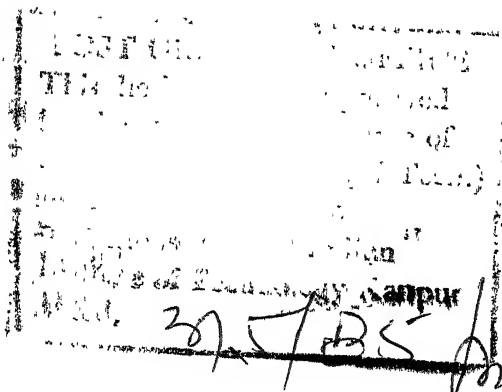
This is to certify that this work entitled, "Glass bonded Ceramic Bodies" by K.N. Rao has been carried out under my supervision and has not been submitted elsewhere for a degree.

B. C. Karpur

Kanpur

Dr. P.C. Kapur
Professor,
Department of Metallurgical Engineering
Indian Institute of Technology, Kanpur

26 April 1985



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- K.N. Rao

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ABSTRACT

Studies were conducted on densification of high refractory and hard filler materials embedded in and bonded with preformed glasses. The glasses were available as wastes. The ceramic bodies with Quartz, Fused Alumina, and Thermit Slag as filler materials and three bonding materials, Pyrex glass, Window glass and E-glass, were examined.

Pyrex glass turned out to be the best bonding matrix, producing dense bodies of 95% Theoretical Density and Tensile Strength of upto 200 kg cm^{-2} . A detailed investigation of the process variables shows that there was optimum fineness for filler. The short firing cycle of 8-10 hours and low firing temperatures of $850-950^{\circ}\text{C}$ required for glass bonded ceramics could result in significant saving in energy.

CHAPTER-I

INTRODUCTION

In conventional ceramic materials like porcelain, vitreous china, steatite or grinding wheel bodies, the microstructure consists of relatively coarse crystalline phase particles embedded in and bonded by a glassy matrix with different levels of porosity. The properties of the ceramic body depend critically on the nature of the microstructure. The normal firing of these materials is carried out at temperatures of 1200 to 1400°C or even more. These high temperatures are required for the formation of a right quantity of molten phase which is formed by decomposition, melting and reaction between the various components of the batch. The energy consumption for this firing step in the manufacture of these ceramic bodies is high and contributes significantly to the cost.

The present work deals with the fabrication of ceramic materials by an energy-saving low temperature vitrification route. The reduction in densification and vitrification temperatures were achieved by exploiting the low softening temperatures of the commercial glasses, which were used as preformed vitreous phase for the bonding matrix. Use of glass also permitted a much shorter firing cycle as sufficient molten phase, required to bind the crystalline components together was

formed directly by phase change, whereas in conventional bodies considerable time is required to form the pyroplastic mass by chemical reactions between the components.

In this work, three types of waste glasses, Window glass, E-glass and Pyrex glass were used as vitreous bonding matrices and three refractory and hard materials, Quartz, Fused Alumina and Thermit Slag were employed as fillers. The different bond-filler combinations were studied for their densification/vitrification behaviour at relatively lower firing temperatures of 700 to 1000°C.

CHAPTER-II

LITERATURE REVIEW

2.1 Sintering, General Considerations

Here, we use the term 'Sintering' in a generic sense, implying thereby a physico-chemical phenomenon comprising of a series of complex processes which lead to consolidation and bonding of particles together in a ceramic body. The progress of the sintering process can be followed by monitoring changes in the physical properties of the body undergoing sintering, eg., porosity, shrinkage/expansion, strength, etc.

There are three stages of consolidation corresponding to three distinct mechanisms of sintering, (a) Viscous flow of liquid resulting in regrouping and compaction of particles, (b) Solution, followed by precipitation leading to mass transfer and (c) solid phase sintering involving solid phase bulk diffusion, or even vapour phase mass transport^{5,11,12,14}. It should be noted that all the three mechanisms need not operate in a given ceramic system. For example, in the firing of almost pure alumina body, no liquid phase is present and the solid phase sintering is the only mechanism which operates. In many classical ceramic bodies such as porcelains, steatites, cordierites, etc., the first two mechanisms, namely, viscous flow and solution precipitation occur concurrently with very

little solid phase sintering, if at all. Whereas in glass bonded ceramics, where the fillers are high refractory materials, such as those which are described in the present investigation, the viscous flow mechanism predominates. A typical densification curve for sintering in the presence of a liquid phase incorporating all the three mechanisms is shown in fig. 2.1⁵.

2.2 Sintering in presence of liquid⁵

In the presence of liquid, as noted above, two mechanisms can operate. In the first stage when the liquid phase is formed, and if the liquid wets the solid particles, the particles are drawn together by capillary forces with simultaneous infiltration of the liquid into the pores. The net result is a reduction in porosity accompanied by shrinkage of the body. The liquid plays a significant role in rearranging and dense packing of particles in the course of consolidation of the body by reducing the inter particle friction. The shrinkage process by this kind of mechanism can occur fairly rapidly and abruptly at the right firing temperatures, which can lead to serious problems of overfiring and slumping. In case the liquid does not wet the particles, it forms isolated pools or globes and contributes little or nothing to densification.

In the second stage, not only the fine particles, but also solid material at sharp edges and corners goes into the solution and is reprecipitated elsewhere leading to mass transfer

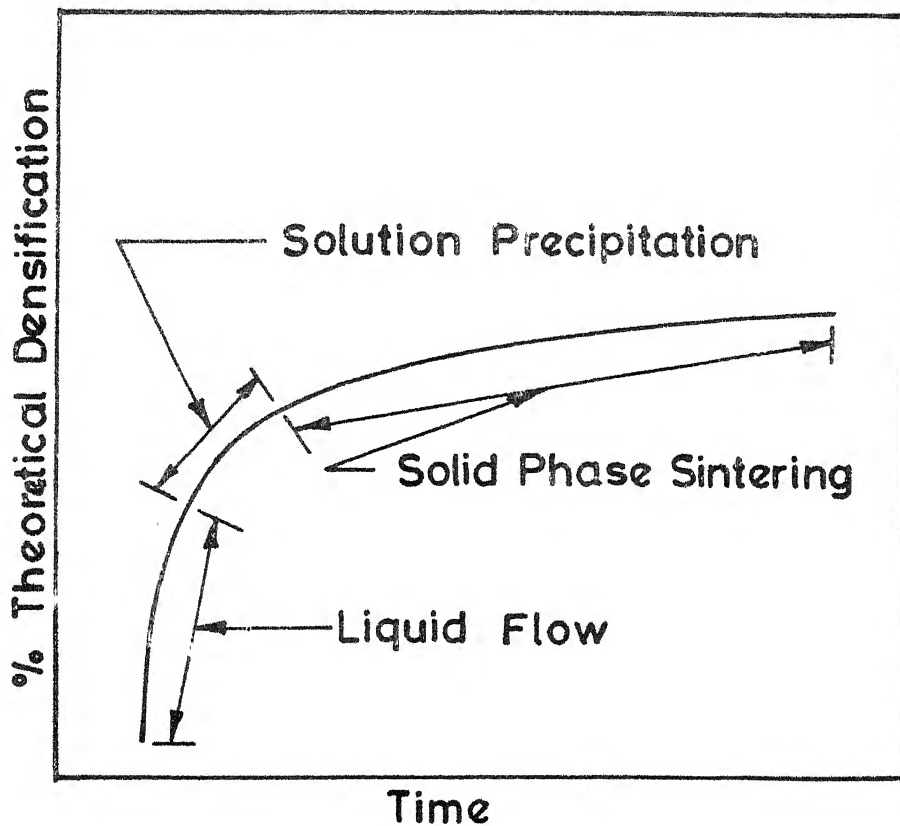


FIG. 2-1 TYPICAL DENSIFICATION CURVE FOR SINTERING IN PRESENCE OF LIQUID PHASE WITH ALL THE THREE MECHANISMS.

and grain growth. This mechanism requires much less liquid than the earlier one, in view of the fact that here the liquid essential function is to provide a conduit for mass transfer as the diffusion rate of species in the liquid phase is substantially higher than in the solid phase. The relative contribution of these two mechanisms is determined by a very complex set of thermodynamic and kinetic considerations and depends on the nature of the system, the phases present and the amount of liquid present.

2.3 Densification and Vitrification by viscous flow mechanism

Since the present work deals primarily with the fabrication of ceramic bodies containing preformed glasses, in which this mechanism predominates, in what follows, the attention will be concentrated on this aspect of sintering. This mechanism of densification and vitrification is most pronounced in those systems where some components are essentially insoluble in the liquid phase formed, at the firing temperature. Such systems are frequently encountered in fabrication of powder metallurgical parts and cermets based on Alumina¹¹.

As mentioned above, this process occurs rapidly once a liquid in sufficient amount having sufficiently low viscosity is formed, resulting in large shrinkages and in general, excellent densification. The net result is a ceramic body having a filler skeleton, embedded in and embounded by a continuous vitreous matrix, with residual pores trapped in the microstructure.

The volume of the pores in the green compact lies in the range of 25 to 50 percent depending on the particle size distribution, their packing and the moulding pressure employed. With sufficient quantity of liquid phase, it is possible to attain the theoretical density by the viscous flow mechanism only which essentially entails regrouping, rearrangement and packing of particles and filling up of the pores by the liquid. According to Cannon and Lenel¹², the quantity of liquid must be at least 25% by volume, on the other hand, Kingery⁷ has calculated that, for spherical filler particles, the minimum quantity of liquid phase for complete densification is 35% by volume. It should be noted that this number is meaningful if the spheres are of single size and pack densely, because only then the packed bed has a porosity of 35%. In fact, Eremenko, et al.⁵, have found that 50% liquid phase is necessary for attaining the theoretical density in real systems. This is understandable because in real systems particles are not only angular and possibly asymmetric, but are also distributed in size and shape.

In presence of insufficient liquid phase, other sintering mechanisms must be superimposed in order to attain complete densification. As said by Kingery⁹, given adequate amount of liquid phase, it is possible to attain complete densification

by viscous flow only and without any change in the grain shape in suitable conditions provided the filler materials exhibit no meaningful solubility in the liquid at the firing temperature.

2.4 Effect of particle size

Kingery⁷ has shown that the degree of vitrification by viscous flow mechanism is inversely proportional to the particle size, whereas in the solution reprecipitation mechanism, it is inversely proportional to the radius to the power $4/3$.

Linel¹² found that the particle size of the filler is a critical parameter in this type of densification mechanism. In Tungsten - Copper system studied by him the extent of vitrification fell markedly when Tungsten powders coarser than 3-5 microns were used. On the other hand with fine powders, although density of green compacts was less, on firing the samples showed greater vitrification and superior mechanical properties. It is hypothesized that the disadvantages of the large size particles are the following, (i) Larger the filler size, greater would be the stresses set up due to thermal expansion/contraction mismatch between the filler and the bonding matrix, (ii) In case of fine size filler, the bonding contact area will be substantially higher, (iii) The apparent viscosity of the pyroplastic mass, mixture of molten phase and the particulate solid, would be significantly higher for the fine size fillers, thereby bestowing considerable mechanical stability to the body.

2.5 Effect of Compacting Pressure

It is well known that fine particles desirable for densification are more difficult to compact mechanically. Therefore high compaction pressures are in general necessary. Plate¹³ noted that the compacting pressure not only reduces the firing shrinkages but also the temperatures of initial interaction between the components. In general higher the compaction pressure, less is the volume of pores that need to be eliminated in vitrification, therefore less is the shrinkage and greater is the dimensional accuracy of the piece.

On the other hand, high compaction pressure can lead to the formation of narrow-necked capillaries or closed pores, due to fracture of particles into ultrafine fragments and even elastic deformation. In firing, considerable gas pressure can build up in these closed pores. As a consequence, this pressure counteracts the capillary pressure and may reduce the rate of flow of liquid into the pores, thereby slowing down the rate of densification. In case the gas pressure exceeds the capillary pressure, the sample may even exhibit expansion or even bloating. In summary, higher compacting pressure has a beneficial effect on sintering provided extensive formation of closed pores is avoided.

2.6 Densification of Glass Powder

The viscous flow mechanism operates when a compact of

powdered glass is sintered as shown by Frenkel³ and, Kuczynski and Zapalatyński⁴. The contribution of mass transfer by the diffusion mechanism seems to be insignificant.

Cutler and Henrichsen² found that crushed glass sinters much more rapidly than spherical glass. The rate of shrinkage depends not only on viscosity but is also inversely proportional to the radius of the particle, because the rate at which the glass softens enough for the flow to occur is inversely proportional to the particle size. And for the same reason, crushed glass particles which have numerous sharp corners or edges would also soften more readily than smooth spherical particles. These authors infact found that the rate of densification of crushed glass was five times that of spherical glass. Hence the particle shape plays an important roll in viscous flow mechanism of vitrification.

2.7 Glass-Filler Bodies

In sintering of porcelain, vitreous china, cordierite temperatures of 1200-1400°C or above are needed inorder to obtain dense and nominally non-porous products. The reason for these high temperatures is that the components in the raw batch mix must react to form a certain amount of molten phase required for densification. The formation of the molten phase occurs usually above 1050°C. It then seems logical to formulate ceramic bodies containing a preformed vitreous phase in a form of powdered

glass in which case the firing temperature and time should be significantly less. Not only this, the glassy components of the raw mix are available as municipal wastes or industrial wastes in sufficiently large quantities.

In a major contribution to this field Wiegmann¹ prepared dense ceramic electrical insulation materials at less than 1000°C. According to him the requirements for successful fabrication of glass-filler type ceramics are the following.

1. The crystalline or filler phase content must be as high as possible in order to assure the maximum strength.
2. For the same reason, the particle size of the filler should be as small as possible consistent with the energy expenditure and contamination in grinding.
3. The fired body understandably should have minimum number of closed pores, although it is not clear how one should go out assuring the absence of these pores except by taking intuitive measures such as particle size control and limit on compaction pressure.
4. Minimise thermal stresses between the glassy matrix and the crystalline phase.
5. Employ glass which softens sufficiently below 1000°C in order to achieve densification at relatively lower temperatures.

As a matter of fact Wiegmann found that the temperature needed to achieve a certain degree of densification increased with increase in the softening point of glass as well as with increase in filler quantity and its particle size. In agreement with the work of cutler and Henrichsen², he also found that firing temperatures are lower if the particle size of the glass is finer.

Wiegmann claims that in firing filler-glass bodies at 850-1000°C, the energy consumption is of the order of 3 to 6 MJ/kg as against 16-25 MJ/kg product needed in conventional fabrication route. Another advantage claimed is the much larger life of the refractory lining in the kiln operating at lower temperatures, as well as higher output due to shorter cycle.

CHAPTER-III

OBJECTIVE OF PRESENT WORK

The filler-glass ceramic bodies seem to be an attractive route for producing a host of ceramic materials at low temperatures in which at least one component, namely glass is a waste material available in large quantity quite cheaply. The central aim of this work was to demonstrate the feasibility of producing glass bonded ceramics at temperatures less than 1000°C using the waste glass available in India for example, ordinary window glass, Pyrex glass and E-glass, in combination with three refractory fillers Quartz, Alumina and another waste product Thermit Slag.

The objective was

1. To study the effect of the following process parameters on the Quartz/Alumina/Thermit Slag based filler-glass ceramic bodies :

- i) Type of glass
- ii) Glass-filler ratio
- iii) Fineness of the filler
- iv) Compaction pressure
- v) Firing temperature

2. To finalise the optimum composition and processing parameters for each of the three fillers from the view point of different applications where the extent of densification varies with products.

CHAPTER-IV

MATERIALS, METHODS AND TESTS

4.1 Materials

The raw materials used were Window Glass (WG), Pyrex Glass (PG), E-glass (EG), Quartz (Q), Alumina (A) and Thermit Slag (T).

4.1.1 Glass

All the glasses used were waste materials. Window glass is a soda lime silicate glass, having relatively low softening points. Pyrex glass is the brand name used in the manufacture of laboratory ware. This is a low expansion borosilicate glass. E-glass is a type of low alkali lime-alumina-borosilicate glass, used in manufacture of glass fibres. All glasses were ground to fine size before use. The chemical composition and other properties of these glasses are given in the Table 4.1, and the viscosity-temperature behaviour is given in the figure 4.1.

4.1.2 Alumina, Quartz and Thermit Slag

White Fused Alumina (+ 99% pure) and Ceramic Quartz, both of -100 mesh size were taken and ground for different times. Quartz occurs in a number of polymorphs. The principal transformations are as follows :

Table 4.1 : Chemical Composition and Properties of glasses

| Property | Window glass | Pyrex glass | E-glass |
|--|--------------|-------------|---------|
| Chemical (wt%) | | | |
| SiO ₂ | 72.0 | 80.5 | 54.5 |
| B ₂ O ₃ | - | 12.9 | 8.5 |
| Al ₂ O ₃ | 1.0 | 2.2 | 14.5 |
| CaO | 11.0 | - | 22.0 |
| MgO | 2.5 | - | - |
| Na ₂ O | 13.0 | 3.3 | 0.5 |
| Others | 0.5 | 0.6 | - |
| Strain Point °C | 515 | 520 | - |
| Anneal Point °C | 550 | 565 | - |
| Softening Point °C | 730 | 820 | 830 |
| True density gm cm ⁻³ | 2.472 | 2.252 | 2.555 |
| Coefficient of Thermal expansion x 10 ⁷ per °C | 85 | 32 | 60 |

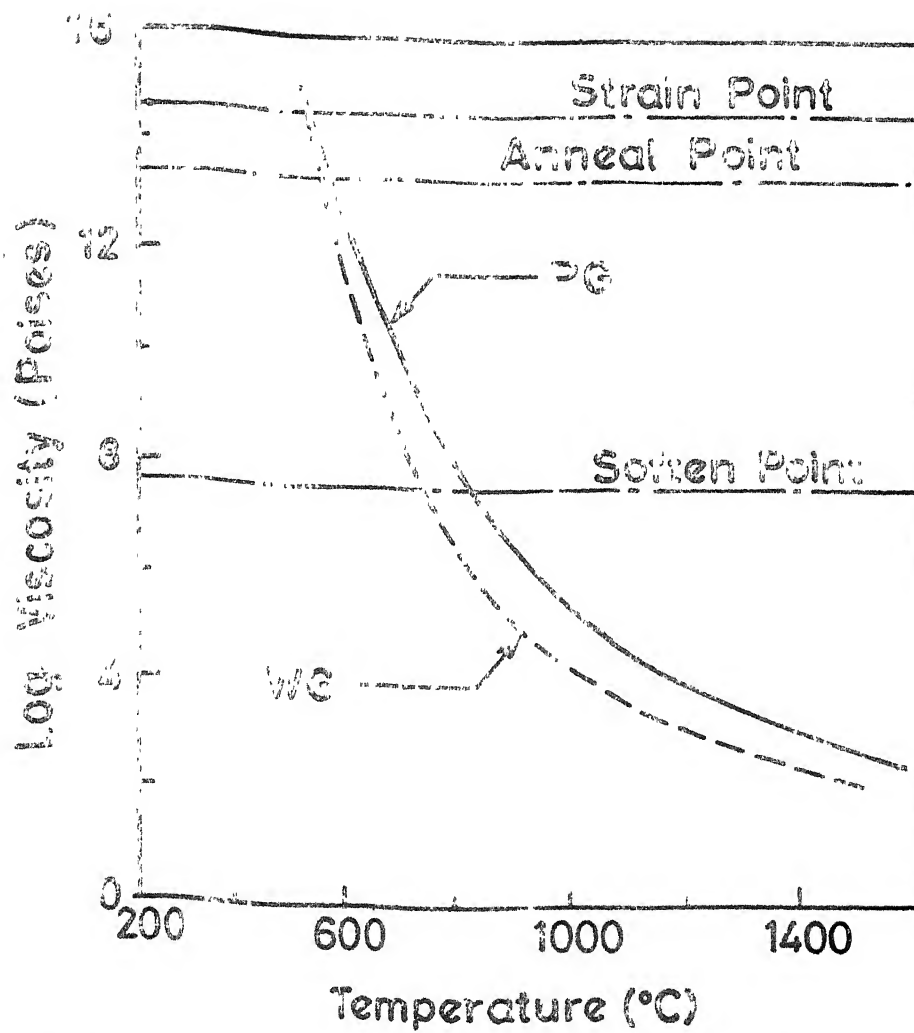
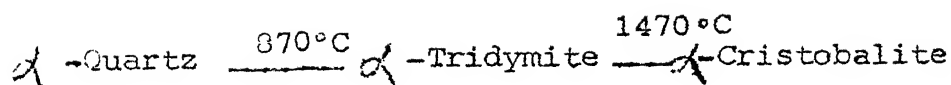


FIG. 4-1 VISCOSITY CURVES OF GLASSES.



In addition, there is a reversible inversion between β -Quartz and α -Quartz at 573°C . Even though the experiments in this work were conducted at below 1000°C , partial transformation of Quartz to higher polymorphs cannot be excluded.

Thermit slag was obtained in lumps from M/S ABM Metalloys Ltd., Shimoga, Karnataka. This was crushed and ground to -100 mesh size for further processing. Its chemical composition in table 4.2 shows that it is a high Alumina waste material with substantial amounts of MnO and CaO.

Table 4.2 : Chemical Composition of Thermit Slag (wt%)

| | | | |
|--------------------------------|-------|-----|------|
| SiO ₂ | 2.10 | CaO | 6.00 |
| Al ₂ O ₃ | 80.30 | MgO | 0.90 |
| MnO | 10.1 | FeO | 0.16 |

The true densities of the three filler materials determined by Pyknometer method are as follows :

| | |
|--------------|---------------------------|
| Alumina | 3.98 gm cm ⁻³ |
| Quartz | 2.68 gm cm ⁻³ |
| Thermit Slag | 3.214 gm cm ⁻³ |

4.2 Methods

4.2.1 Fine Milling

All the raw materials were fine ground separately. The waste glasses were washed clean and first broken to -5mm size in an ordinary cast iron mortar and pestle. The crushed glass was cleaned again and then wet ground in a ball mill. The filler materials being already-100 mesh size, were wet ground directly. The glasses were ground for 25 hours, and the three filler materials for 10, 20 and 40 hours. The specifications of the ball mills were as follows :

Ball mill for grinding filler

| | |
|--|----------|
| Height | 120 mm |
| Internal diameter | 121 mm. |
| External diameter | 143 mm |
| Useful volume | 1.14 lit |
| Revolutions per minute | 115 |
| Weight of filler taken | 250 gm |
| Weight of high alumina grinding media of size 12.5x12.5 mm cylinders | 1 kg |

Ball mill for grinding glass

| | |
|-------------------|--------|
| Height | 200 mm |
| Internal diameter | 210 mm |
| External diameter | 232 mm |

| | |
|---|---------|
| Useful volume | 6 lit |
| Revolutions per minute | 72 |
| weight of high alumina grinding media of size | |
| 20x20 mm cylinders | 2.25 kg |
| 12.5x12.5 mm cylinders | 2.25 kg |
| Weight of glass taken | 1.50 kg |

The quantity of water varied with different materials.

225 ml per 250 gm of Quartz taken

250 ml per 250 gm of Alumina taken

400 ml per 250 gm of Thermit Slag taken

2 l per 1.5 kg of glass taken.

4.2.2 Pyrometric Cone Tests of Filler-Glass Compositions

For preliminary screening and to obtain a qualitative idea of the softening and flow behaviour of various filler-glass compositions, these mixtures were made into standard laboratory size cones and subjected to pyrometric cone tests, i.e. the temperatures at which the cones initially tipped, half tipped and fully tipped were noted.

Cone making and mounting : Ground filler and glass in different proportions were thoroughly mixed and 4% dextrin was added. The wet mix was moulded into the shape of cones in a steel mold and dried in an oven at 80°C. Lower percentage of dextrin did not provide enough green strength whereas higher dextrin contents lead to cracking of the cones in firing. These dry cones were mounted on a circular alumina plate called plaque which had

suitable grooves cut in the periphery for the purpose. Refractory clay was used to cement the cones in place. An inward slant of about 10° to the vertical was maintained for all the cones.

Size of the cones

1. Overall height (between planes perpendicular to the triangular axis) = 28.56 mm
2. Length of the sides of the triangular tip = 1.91 mm
3. Length of the sides of the base = 7.14 mm

Tipping of cones : The plaque with the cones was heated uniformly at a rate of 2°C min and the temperatures at which the cones started softening i.e. began to tip, bent one half and fully tipped were noted down by means of a Pt-Rh thermocouple placed in the centre of the plaque.

4.2.3 Pellet making :

Small Pellets : Pellets of diameter 12.5 mm and thickness ranging from 3 to 8 mm were made in a three-piece split die. Two drops ($1 \text{ drop} \approx 0.05 \text{ cm}^3$) of 5% dextrin solution was added to each gram of dry powder mix, for providing adequate green strength to the pressed pellets. After thoroughly mixing the filler and glass powders in a porcelain mortar and pestle, the mixture was moulded in a hydraulic press at any of the two level of pressures, 375 kg cm^{-2} or 1500 kg cm^{-2} . Adequate cleaning of die with Acetone and lubrication with Liquid Parafin was

practised. The pellets were dried for more than a day at around 100°C before firing. These pellets were used to monitor the densification of the body.

Big pellets : For strength testing, bigger samples of 25 mm diameter and 8 to 12 mm thick were pressed. Now dextrin was not used as to permit a faster rate of firing. Only a few drops of water were enough to give adequate green strength for handling after pressing at 1500 kg cm⁻². The big samples were thoroughly dried for 30 hours at 100°C. As some surface cracks showed up after drying in many samples, these were removed by carefully grinding the dry green pieces on a belt grinding machine so as to remove about 2 mm of the thickness. Each sample was marked, its bulk density was determined and then fired.

4.2.4 Firing

Pellets were fired in a tube furnace of diameter 5 cm. The pellets were loaded one high on a bed of coarse alumina particles. For big pellets a heating rate of 5°C min⁻¹ was used. On account of the presence of dextrin in small pellets, slower heating rate of about 3°C min⁻¹ was adapted for these. Samples containing quartz were also heated at a slower rate specially in the 550-600°C range where β -Quartz to α -Quartz transformation occurs. The pellets were soaked at the maximum temperature for one hour for small and two hours for big pellets, after which they were furnace cooled to room temperature. Typical firing

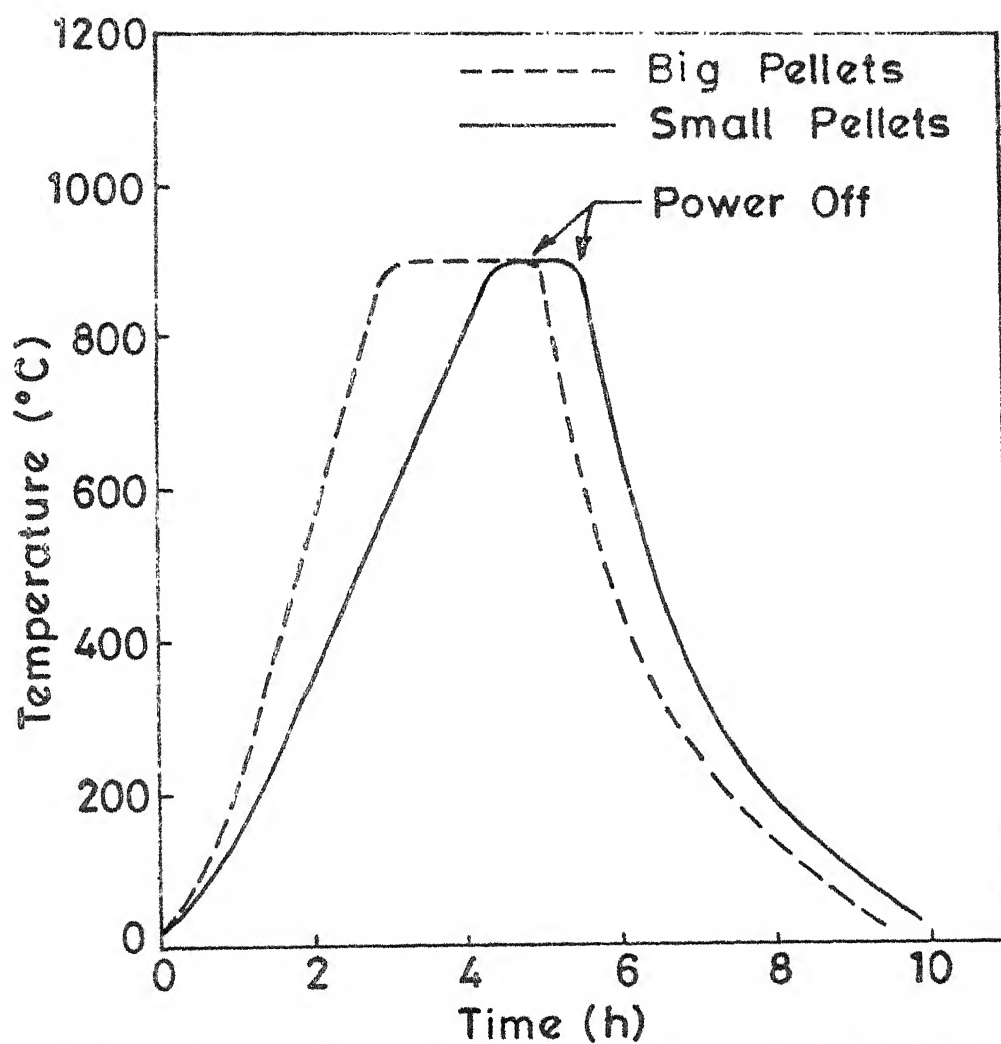


FIG. 4-2. FIRING CYCLES FOR PELLETS.

curves are shown in fig. 4.2. Same set of small pellets was used to get densification data at different firing temperatures, i.e. 700, 750, 800, 850, 900, 950 and 1000°C.

4.3 Tests

4.3.1 True Density of Powders

True densities were determined by the pyknometer method. Water was used as the displacement liquid. Care was taken to remove any air bubbles trapped in the powder. The pyknometer bottle with the powder and half filled with water was maintained at 100°C in a boiling water bath for around 10 minutes so that the entrapped air bubbles were driven out. The weight measurements were taken after the pyknometer and its contents cooled down to room temperature. Data on the densities of all the materials are given in Appendix I.

4.3.2 Bulk Density of Pellets

Bulk densities of small pellets were measured using the mass and volume of the pieces; the latter was measured by micrometer. Bulk densities of big pellets were measured by water displacement. The sample was weighed in air, boiled in water for half an hour, weighed under water, and then weighed while still soaked with water, in air.

4.3.3 Surface Area of Powders

Specific surface areas of all the powders were determined using Blaine's apparatus and a standard sample of Portland Cement.

The method involves preparation of a uniformly packed bed of known porosity (usually 50%) in a standard cell and noting the time taken for a fixed volume of air at constant temperature to pass through this bed.

The formula is

$$S = S_s \frac{\rho_s (1-e_s)}{\rho (1-e)} \sqrt{\frac{e^3}{e_s^3} \cdot \frac{\eta_s}{\eta}} \sqrt{\frac{T}{T_s}}$$

where,

S = Specific surface area in $\text{cm}^2 \text{ gm}^{-1}$ of the test sample.

S_s = Specific surface area in $\text{cm}^2 \text{ gm}^{-1}$ of standard sample used in calibration of the apparatus. Here ordinary Portland Cement was used ($S_s = 2250 \text{ cm}^2 \text{ gm}^{-1}$)

T = Measured time interval in seconds of manometer drop for test sample.

T_s = Measured time interval in seconds of manometer drop for sample used in the calibration of apparatus.

η = Viscosity of air in poise at the temperature of test of the test sample.

η_s = Viscosity of air in poise at the temperature of test of the standard sample used in calibration of apparatus.

e = Porosity of prepared bed of test sample

e_s = Porosity of prepared bed of standard sample
(Portland Cement)

- ρ = True density of test sample in gm cm^{-3}
 ρ_s = True density of standard sample used in calibration
 of apparatus (Assumed to be 3.15 gm cm^{-3} for
 Portland Cement)

When the measurements are done at the same temperature, the formula simplifies to

$$S = S_s \frac{\rho_s}{\rho} \frac{(1-e_s)}{(1-e)} \sqrt{\frac{e^3}{e_s^3}} \sqrt{\frac{T}{T_s}}$$

Specific surface area values of all the materials are given in the Appendix I.

4.3.4 Splitting test :

The big disc shaped pellet was placed between the platens of the Instron testing machine such that its axis was horizontal as shown in the figure 4.3. The load was increased at a rate of 0.05 mm min^{-1} cross head speed until failure occurred by splitting along the vertical diameter of the disc. When the load is applied along the generatrix, then an element near the end of vertical diameter of the cylinder is subjected to a compressive stress of

$$\frac{2P}{\pi HD} \left[\frac{D^2}{r(D-r)} - 1 \right]$$

and a horizontal tensile stress of

$$\frac{2P}{\pi HD}$$

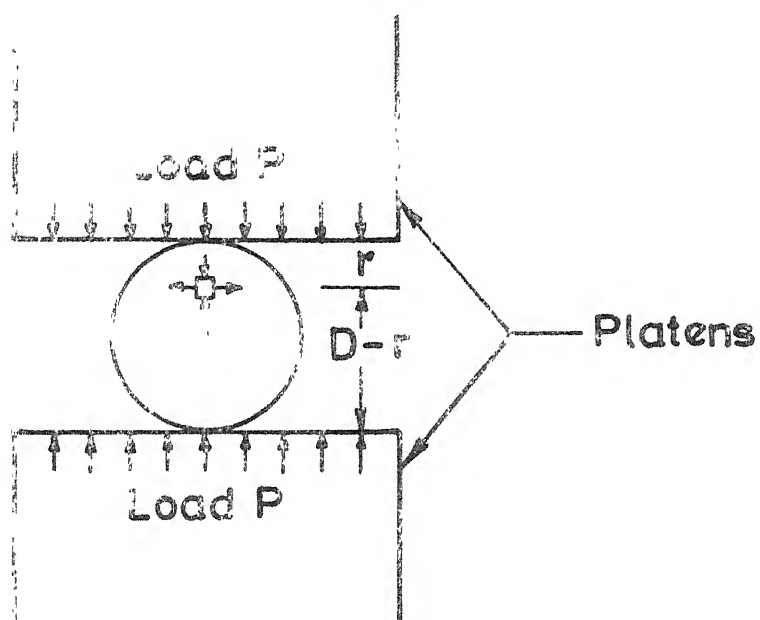


FIG. 4-3. CONFIGURATION OF SPLITTING TEST.

where P is the compressive load on the disc

H is the height of the disc

D is the diameter of the disc

The splitting occurs due to this tensile stress and hence the tensile strength of the sample can be calculated from above.

CHAPTER-V

EXPERIMENTAL RESULTS

5.1 Grinding of Raw Materials

The filler materials were ground for 10, 20, 40 hours and the glasses for 25 hours as described in section 4.2.1. The specific surface areas of the powders as a function of grinding time, are shown in figure 5.1. It can be seen that, as expected, increment in fineness decreases as the time of grinding increases because the energy required for grinding the materials becomes disproportionally high as the particles become finer.

5.2 Preliminary Experiments

As the aim was to produce ceramic materials at relatively low firing temperatures, the first experiments were conducted to estimate the probable vitrification temperature range, by the pyrometric cone tests. The tipping temperatures of test cones subjected to a constant rate of heating are shown in figures 5.2 and 5.3.

The first figure shows Window Glass-Quartz compositions in which the data is exhibited in three separate boxes for 10 h, 20 h, and 40 h ground Quartz. Each box has three curves for initial tipping, half bent and fully tipped temperatures as a function of percentage bond by volume. This data is plotted again in the second figure in which, each box pertains

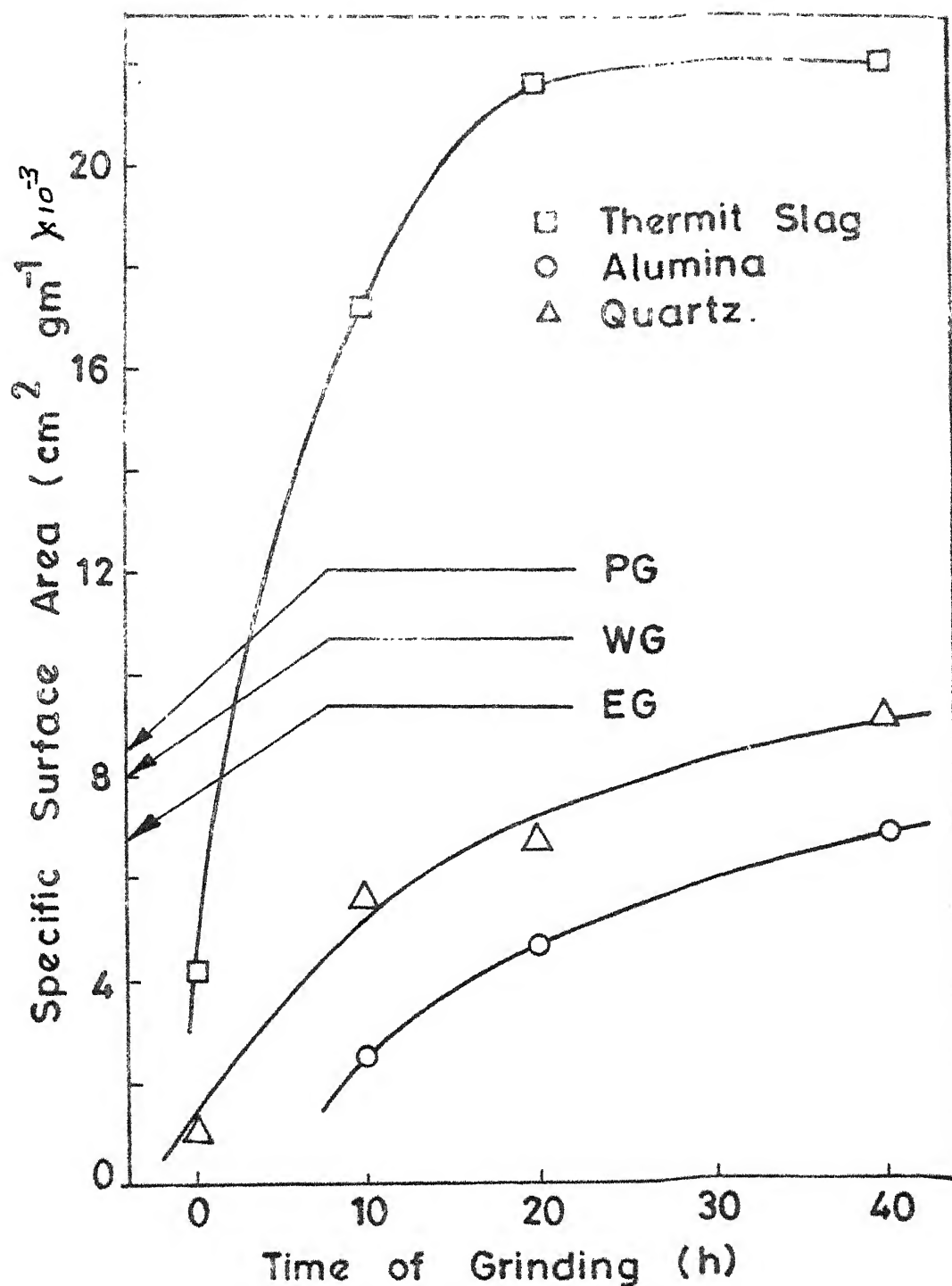
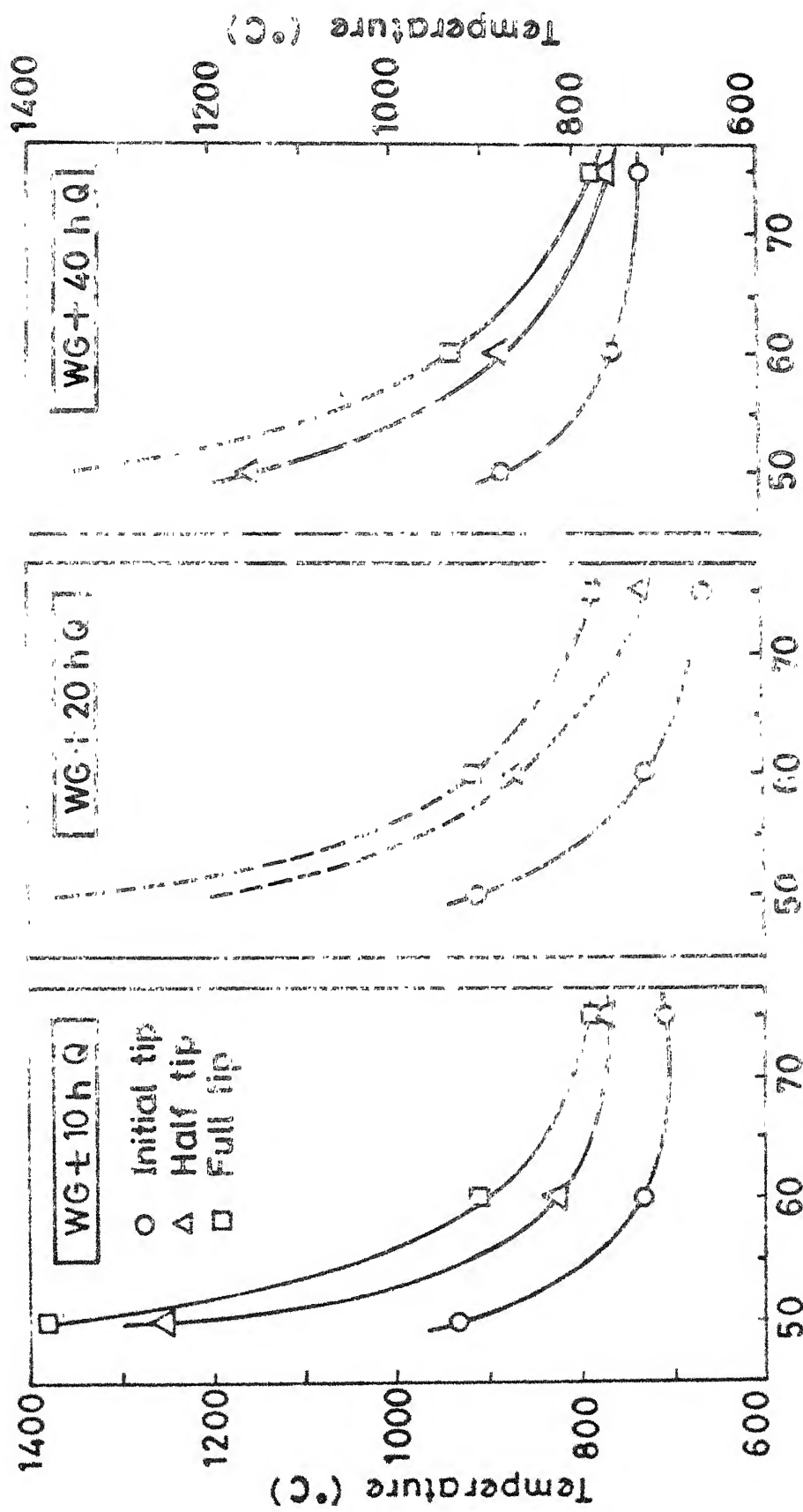


FIG. 5-1. INCREASE IN SPECIFIC SURFACE AREA WITH GRINDING TIME.



Glass Content in Glass-Quartz Compositions (% Volume)

FIG.5-2. TEMPERATURES OF INITIAL TIP, HALF TIP, AND FULL TIP OF PYROMETRIC TEST CONES MADE OF DIFFERENT WINDOW GLASS-QUARTZ COMPOSITIONS.

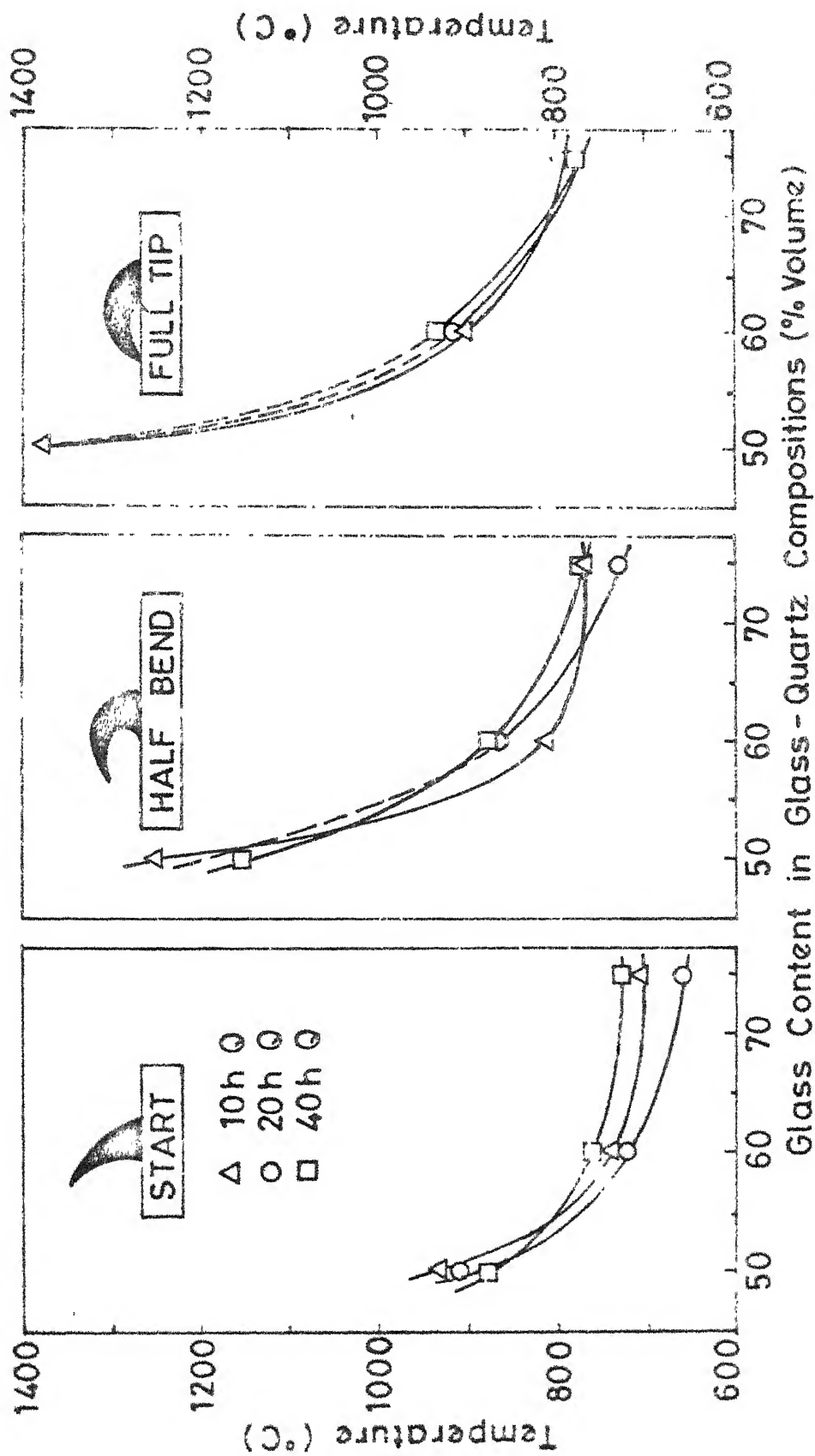


FIG. 5-3. SOFTENING POINTS OF TEST CONES OF DIFFERENT WINDOW GLASS -QUARTZ COMPOSITIONS.

to the state of cone namely initial tipping, half tip and fully bent.

It will be noted that in general between 50% to 60% bond by volume, there occurs a sharp drop in temperature for all three states of cone tipping. Between 60 and 75% bond by volume, the drop in temperature is not so sharp. It also seems that for the temperatures of fully tipped cones the fineness of Quartz is not a matter. But there are significant differences for the temperatures corresponding to initial and half tipping. Comparison with latter experiments on pellets lead one to believe that temperatures in the vicinity of initial tipping are the required firing temperatures for maximum densification attainable.

In view of large amount of time needed for carrying out the pyrometric cone test, it was decided at this stage to proceed directly to the pressed pellets. Further it was decided to use the finest powders available and hence further experiments were conducted with only 40 h ground Quartz, Alumina and Thermit Slag.

5.3 Selection of Vitreous Bonding Matrix

Each filler was bonded with three types of glasses, in 60 and 50% volume of glass. Thus in all 18 compositions regulated. Pellets of 1.25 cm diameter were pressed under 375 kg cm^{-2} pressure and fired in the range of 700 to 1000°C. The results are exhibited by figures 5.4, 5.5 and 5.6 for Quartz, Alumina and

Slag respectively.

Figure 5.4, shows the percentage of theoretical density (% TD) attained as a function of firing temperature for 6 compositions in which three glasses at 60% and 50% volume levels each were used. It will be seen that 60% Pyrex Glass and 50% Window glass, gave the best results. With 60% Window glass, the samples showed very poor densification. In general, as expected, higher the percentage of glass content, higher was the relative theoretical density attained at a given firing temperature.

Figure 5.5 for Alumina similarly shows the marked superiority of 60% volume of Pyrex glass over all other bonds, resulting in a densification of 92% of theoretical density at 1000°C. Interestingly enough with Window glass at both 50% and 60% levels, the percentage theoretical density attained is invariant of temperature in the range of 800-1000°C. Another noteworthy feature is the very large difference in the densification results between 60% and 50% bonds for Pyrex glass. Again the E-glass showed disappointing results as far as densification is concerned.

Figure 5.6 for Thermit Slag again shows the superiority of Pyrex glass at 60% levels over other bonds, plus the fact that the maximum relative theoretical density reached is invariant of the firing temperature over the range 850 to 950°C.

To summarise these results, under the experimental conditions obtaining, Pyrex glass is by far the best bonding matrix.

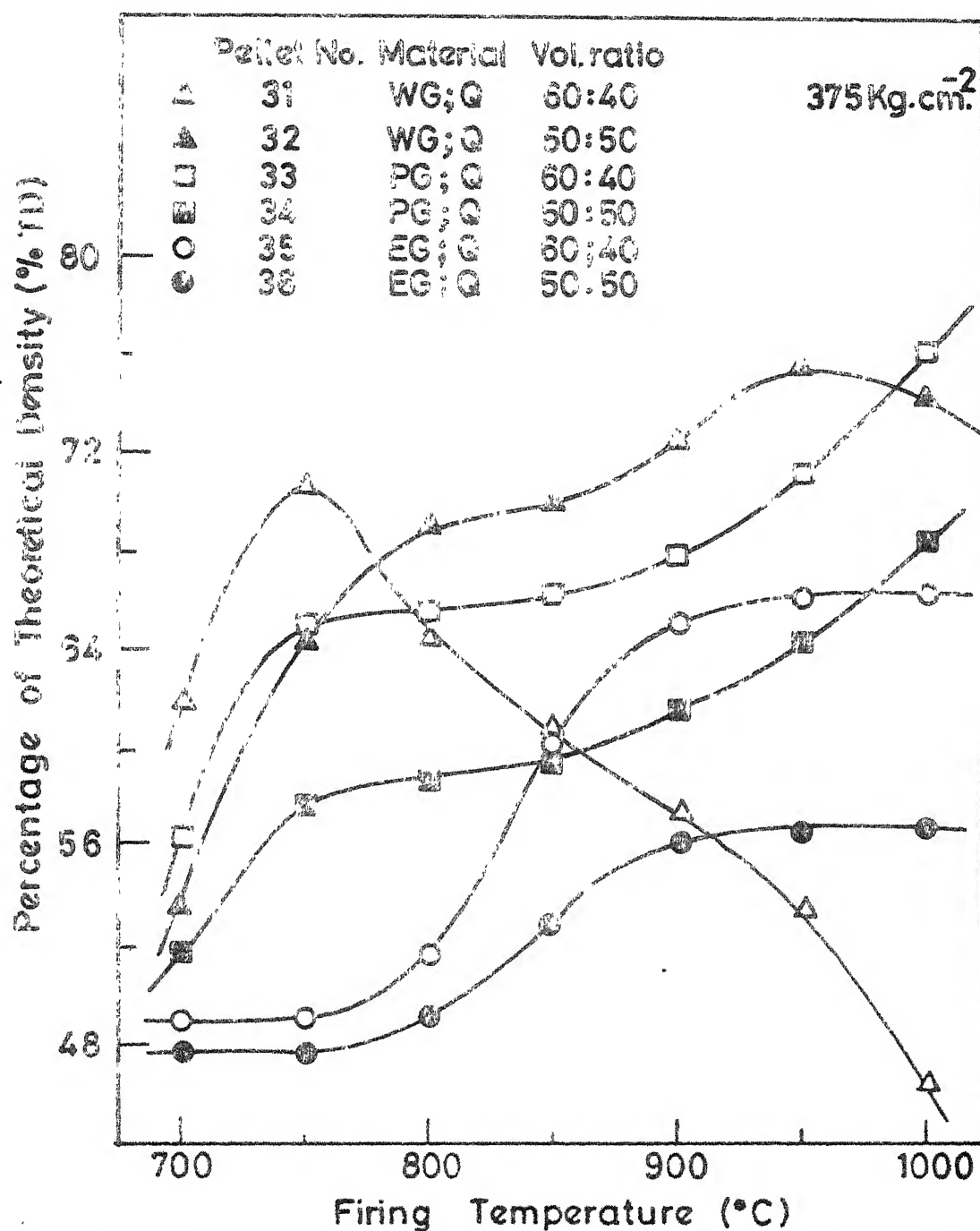


FIG. 5-4. PERCENTAGE OF THEORETICAL DENSITY ATTAINED AT DIFFERENT FIRING TEMPERATURES FOR 40h GROUND QUARTZ BONDED WITH 3 GLASSES.

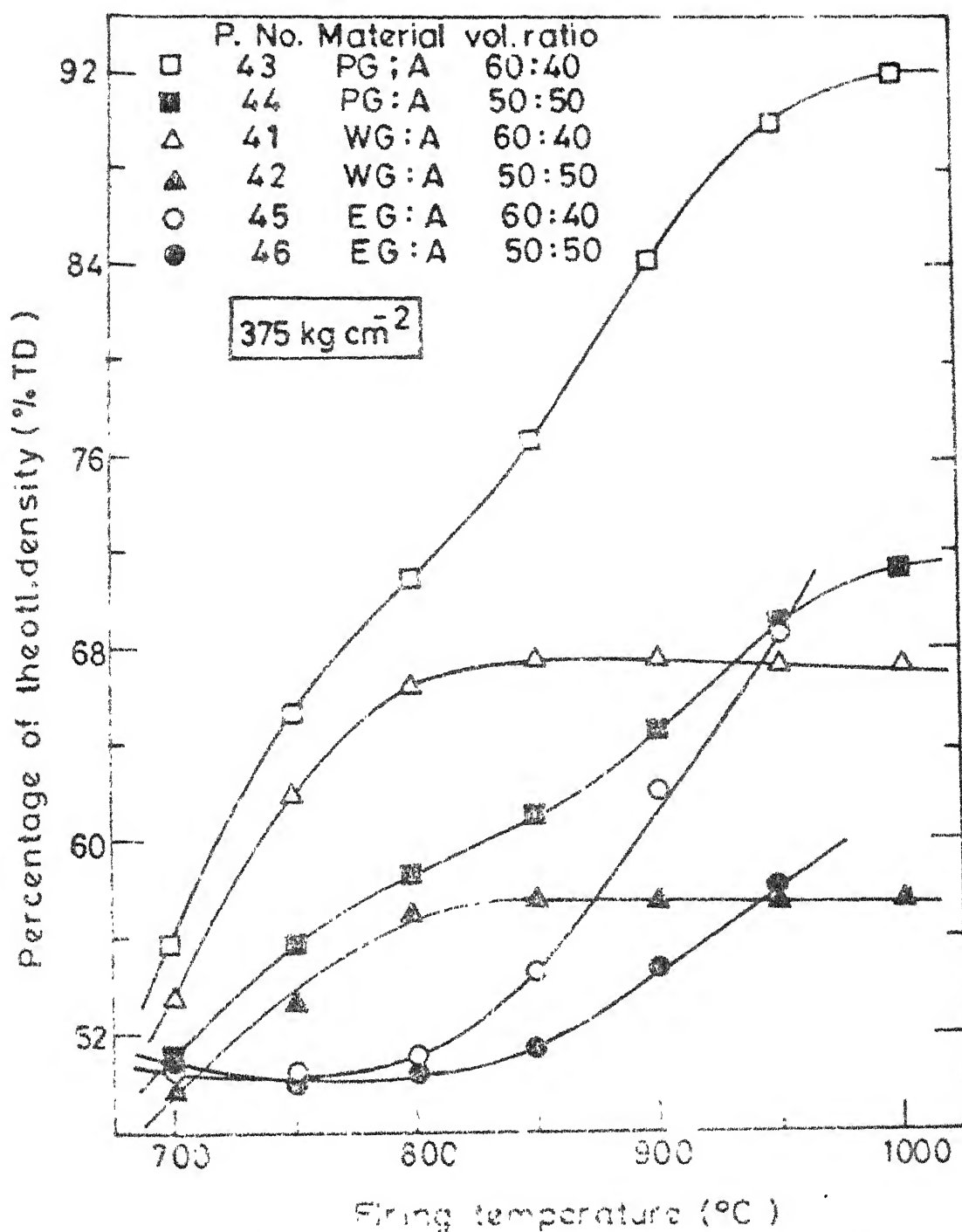


FIG 5-5 PERCENTAGE OF THEORETICAL DENSITY ATTAINED AT DIFFERENT FIRING TEMPERATURES FOR 40% GROUND ALUMINA BONDED WITH 3 GLASSES

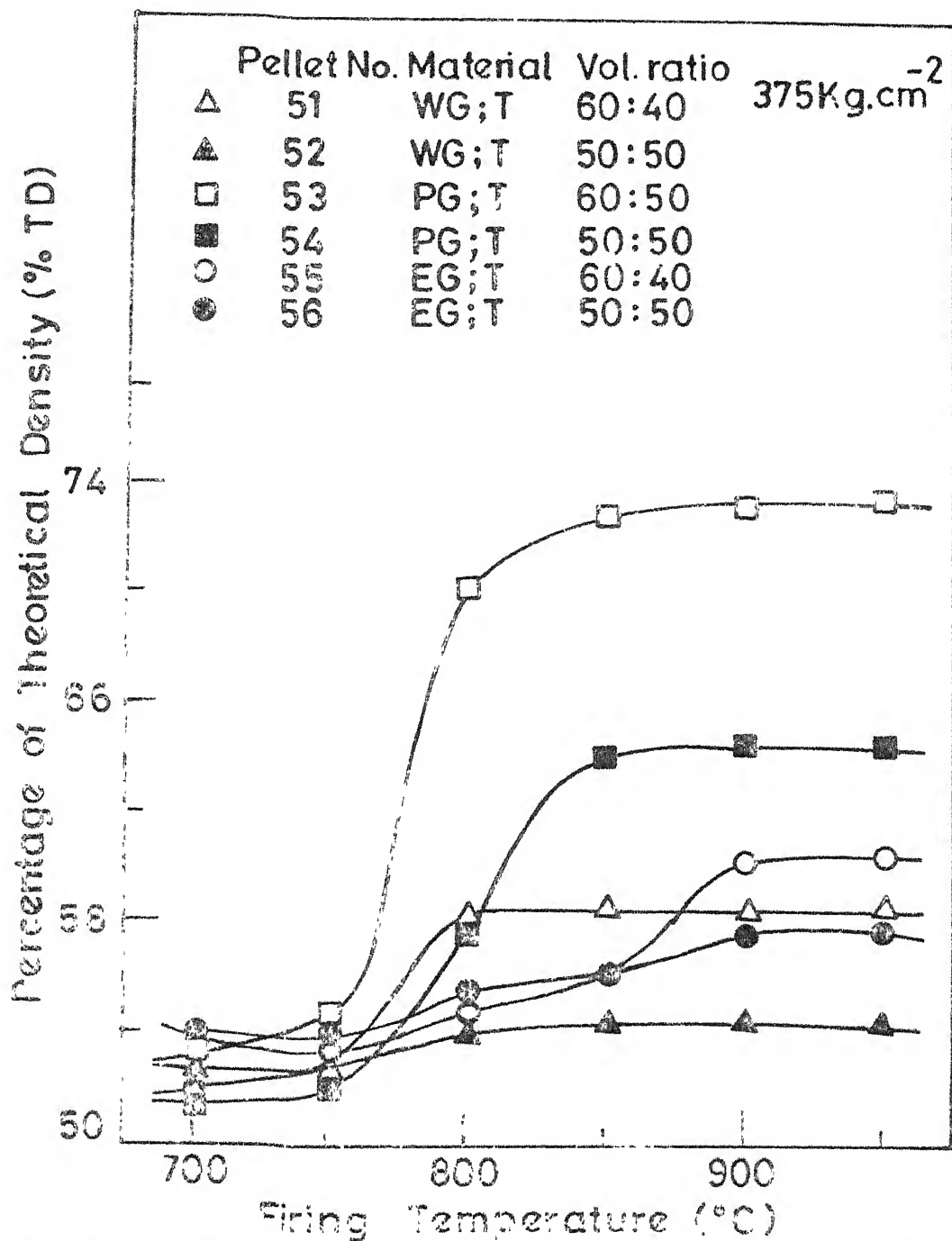


FIG. 5-6. PERCENTAGE OF THEORETICAL DENSITY ATTAINED AT DIFFERENT FIRING TEMP. FOR AL₂O₃ GROUND SLAG BONDED WITH 3 GLASSER

It is possible to attain densification of 90% plus, for Alumina and about 75% for Quartz and Thermit Slag with 60% volume of Pyrex glass.

5.4 Enhancement of Densification

From the above results, for two fillers Quartz and Thermit Slag, the densification achieved was only of the order of 75% and needed to be enhanced further. For this purpose two changes were made i) increase in the moulding pressure from 375 kg cm^{-2} to 1500 kg cm^{-2} and ii) increase in Pyrex glass bond content from 60 to 65 and then to 70% by volume. The results are shown in figures 5.7, 5.8 and 5.9 for Quartz, Alumina and Thermit Slag respectively, along with earlier results with 60% volume bond and moulding pressure of 375 kg cm^{-2} .

Fig. 5.7 shows, as expected, that the densification improved with increase in moulding pressure as well as with higher bond content and with 1500 kg cm^{-2} moulding pressure, it was possible to attain 90% of theoretical density. This calculation is done on the assumption that the Silica remains in the form of Quartz with density of 2.68 gm cm^{-3} . It is however possible that with the thermal treatment done, some quartz has undergone transformation to higher polymorphs, Tridymite and Cristobalite which have significantly lower densities. If this indeed had happened, the achieved densification obtained would be somewhat higher, perhaps as high as 95%.

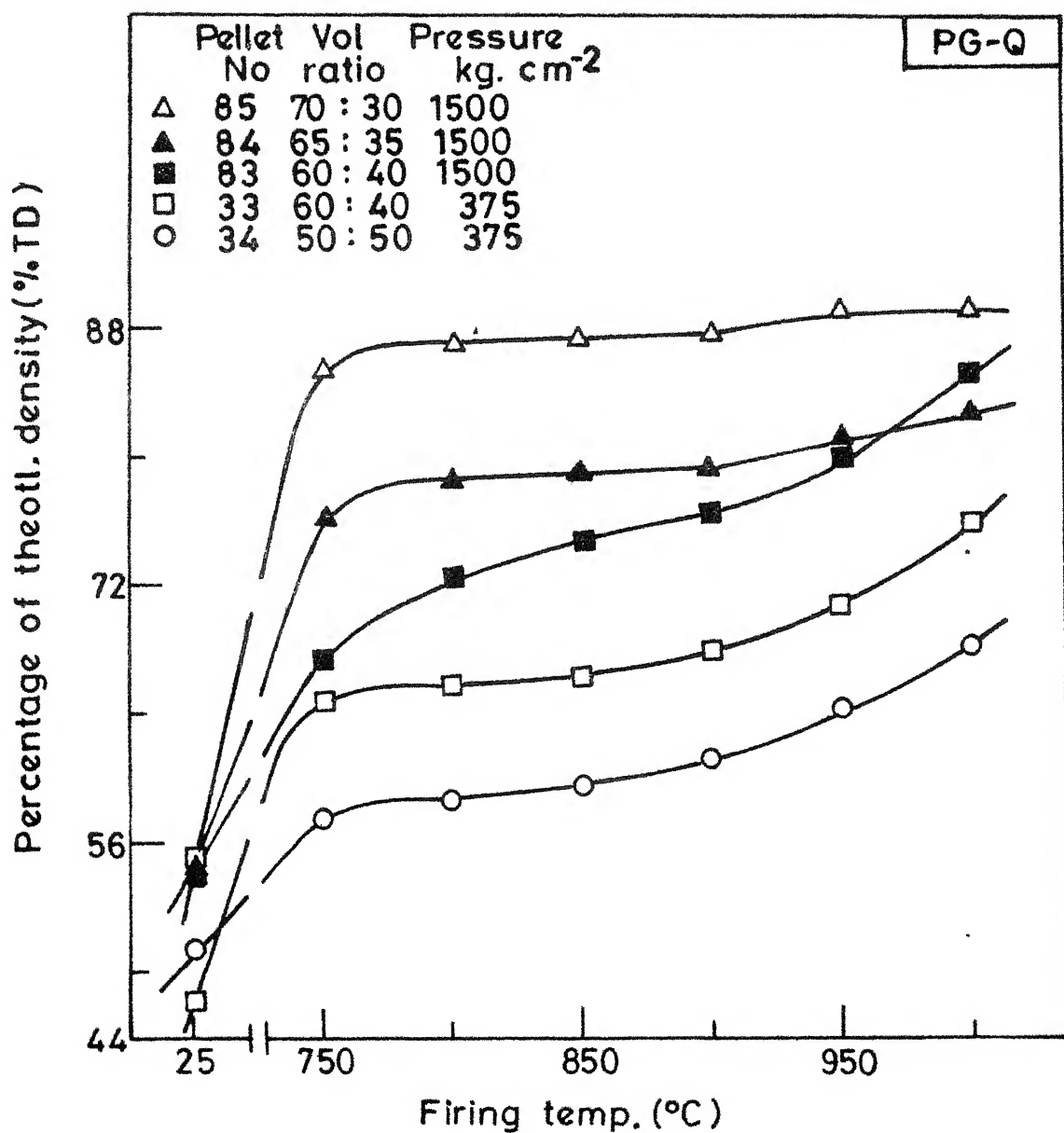


FIG. 5.7 PERCENTAGE OF THEORETICAL DENSITY AS A FUNCTION OF FIRING TEMPERATURE FOR PYREX - QUARTZ (40h) PELLETS

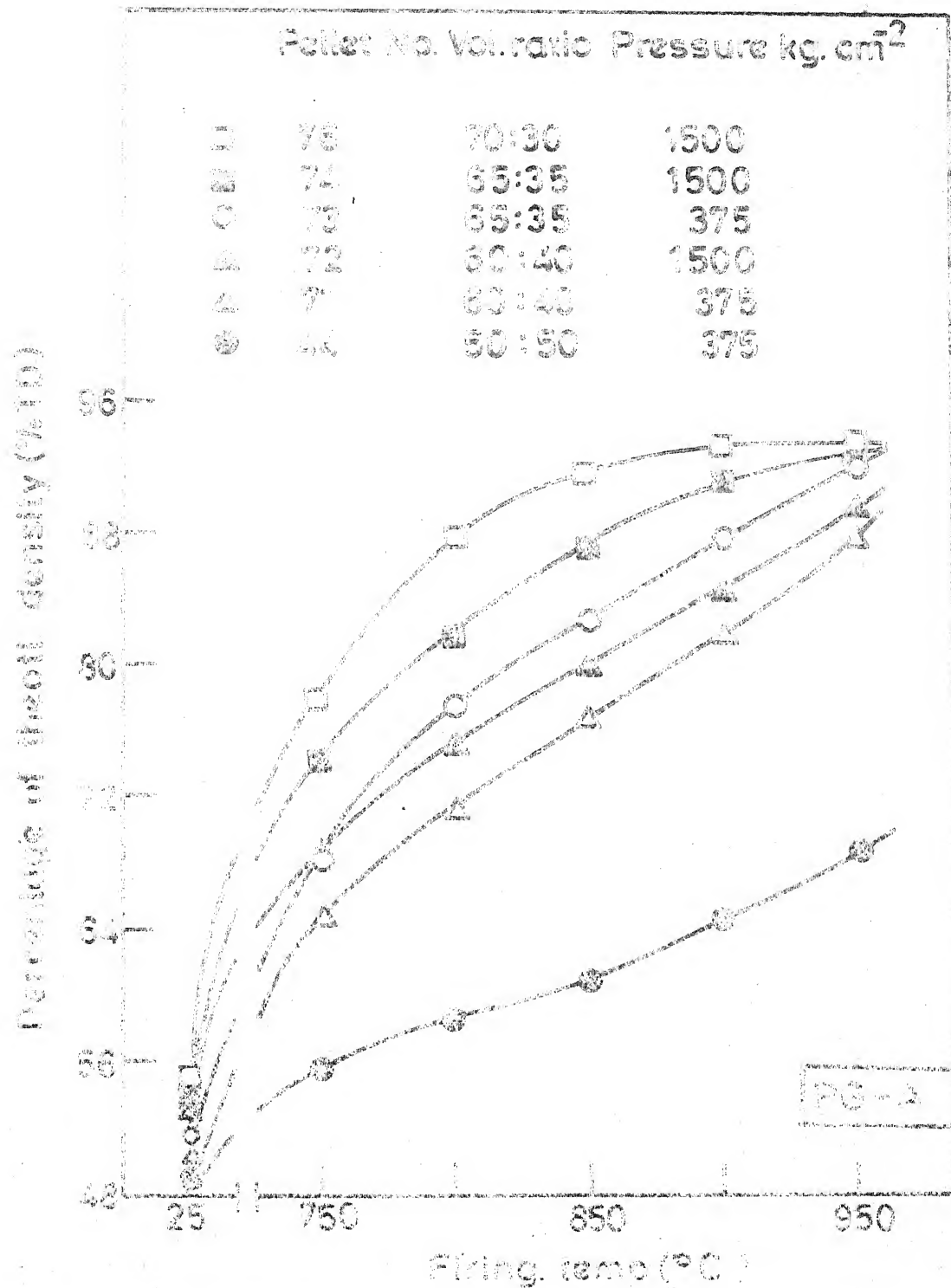


FIG 5.8 PERCENTAGE OF THEORETICAL DENSITY AS A FUNCTION OF FIRING TEMPERATURE FOR PYREX ALUMINA (40h) PELLETS

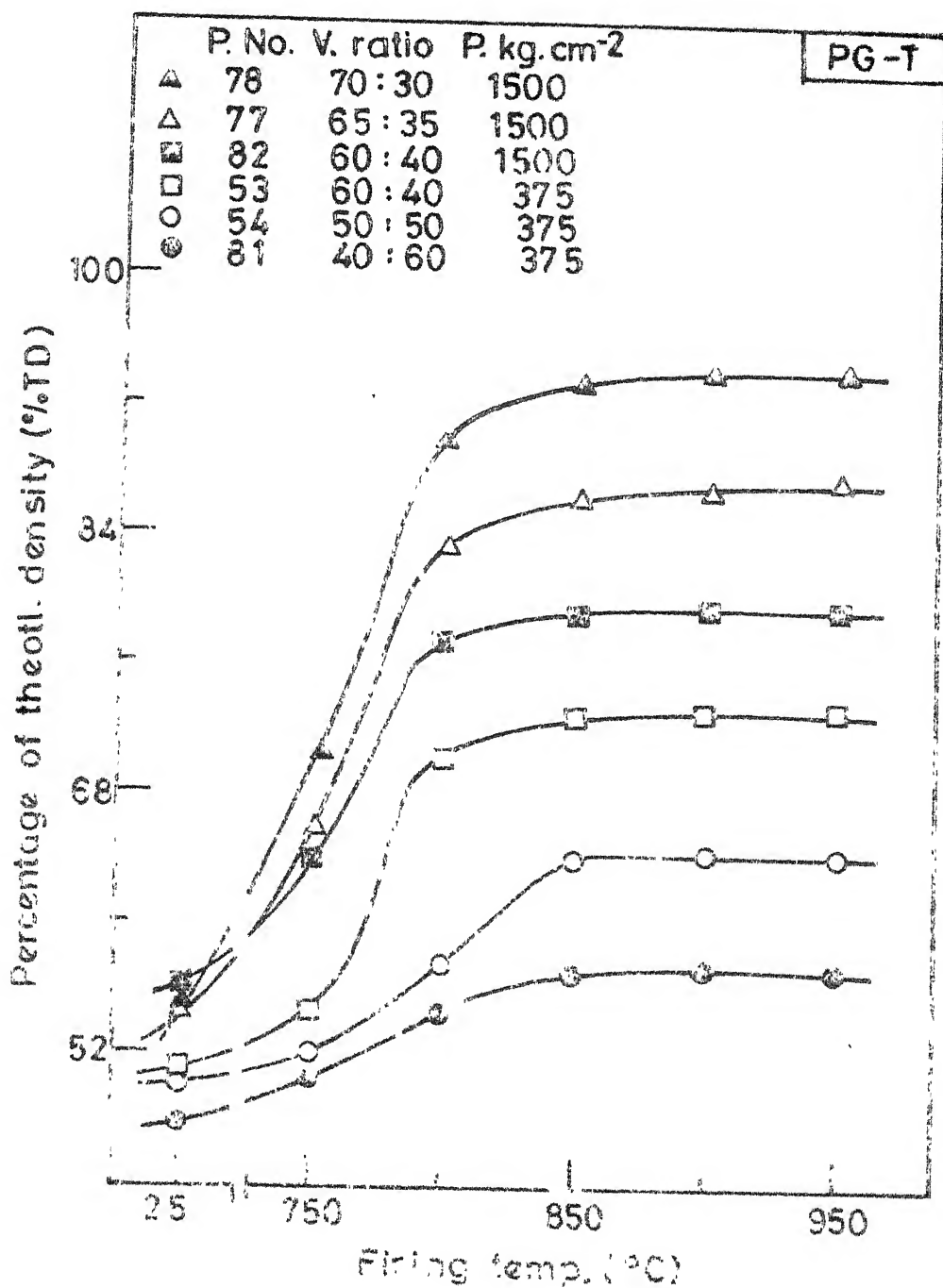


FIG 5.9 PERCENTAGE OF THEORETICAL DENSITY AS A FUNCTION OF FIRING TEMPERATURE FOR PYREX-THERMOT SLAG (406) PELLETS

Fig. 5.8 shows that unlike Quartz, in Alumina-Pyrex glass system, the sensitivity to bond content as well as pressure is not so marked, specially if the bond content exceeds 65% volume and firing temperatures are around more than 900°C. In this case, densification of the order of 94% of theoretical density can be reached at 900°C.

In case of Thermit Slag in figure 5.9, the results are similar to those of Quartz in figure 5.7. Here again densification of 94% of theoretical density could be attained using 70% volume of bond and 1500 kg cm^{-2} moulding pressure at temperatures of 850°C.

5.5 Effect of Filler Fineness

Although it has been claimed in literature that, finer the size of the filler particles, better are the results, it should be appreciated that grinding is a highly energy intensive unit operation, specially for hard refractory materials such as Alumina and in size ranges of interest i.e. less than 40 microns. In order to confirm whether very fine grinding is really necessary, the best compositions obtained in the previous set of experiments (i.e. 70% bond and 1500 kg cm^{-2} moulding pressure) were repeated using fillers ground for 10 and 20 hours. The results are shown in figures 5.10, 5.11, 5.12 for Quartz, Alumina and Thermit Slag respectively. Surprisingly, coarser powders obtained by 10 hours grinding gave in all three cases

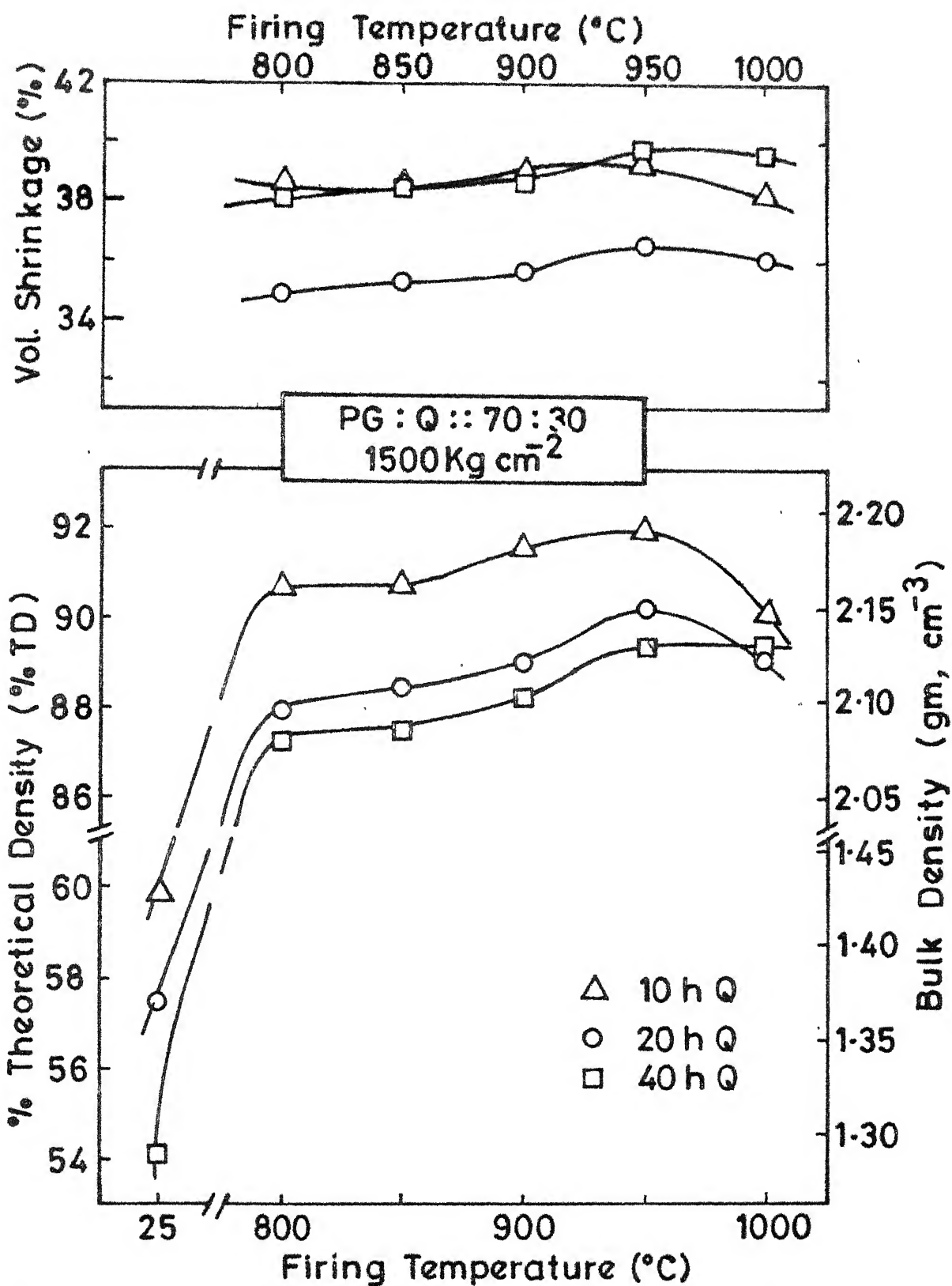


FIG. 5-10. SHRINKAGE, % TD, BULK DENSITY OF PYREX-QUARTZ PELLETS AT DIFFERENT FIRING TEMPERATURES.

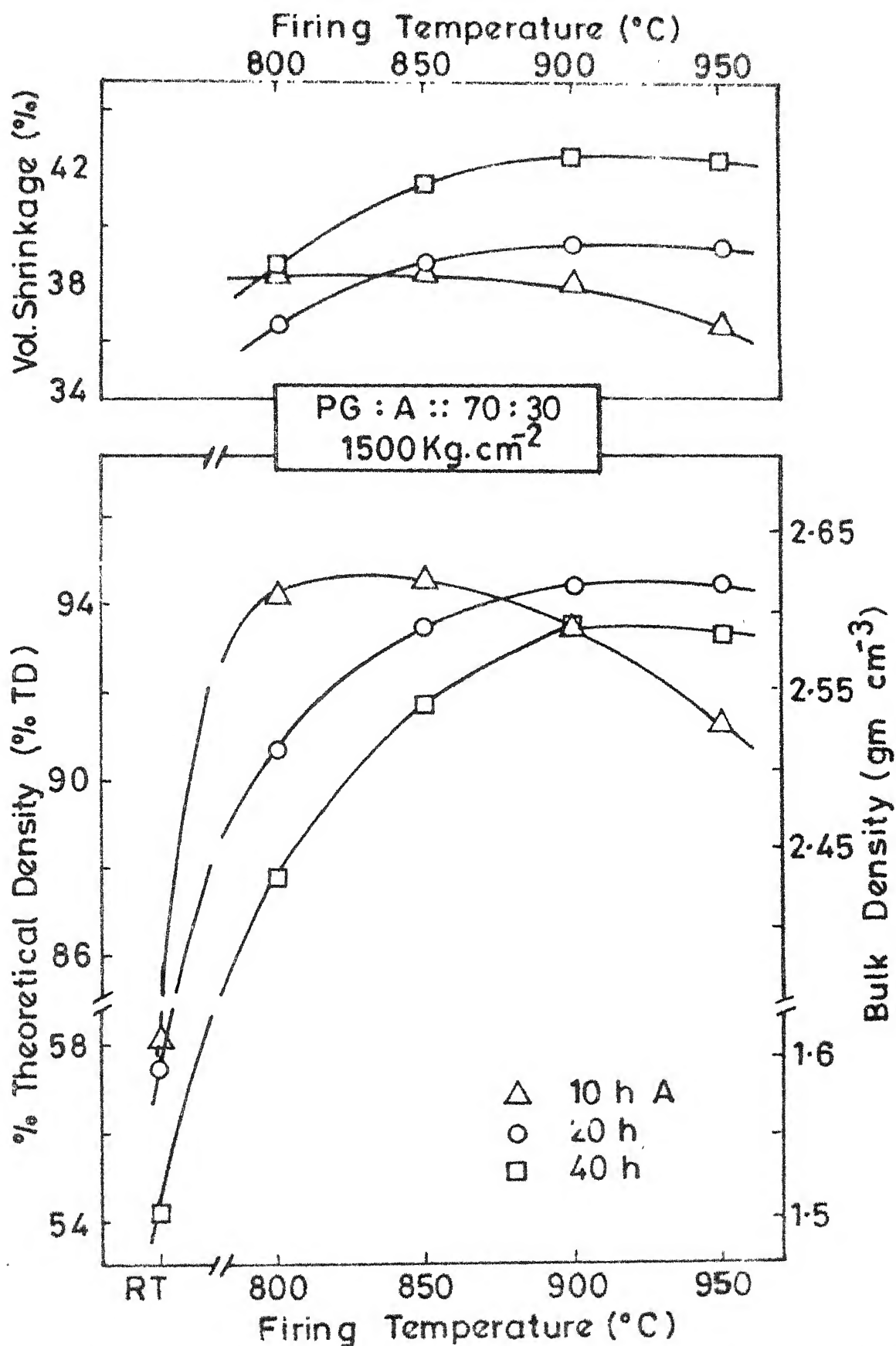


FIG. 5.11. SHRINKAGE, % TD, BULK DENSITY OF PYREX - ALUMINA PELLETS AT DIFFERENT FIRING TEM

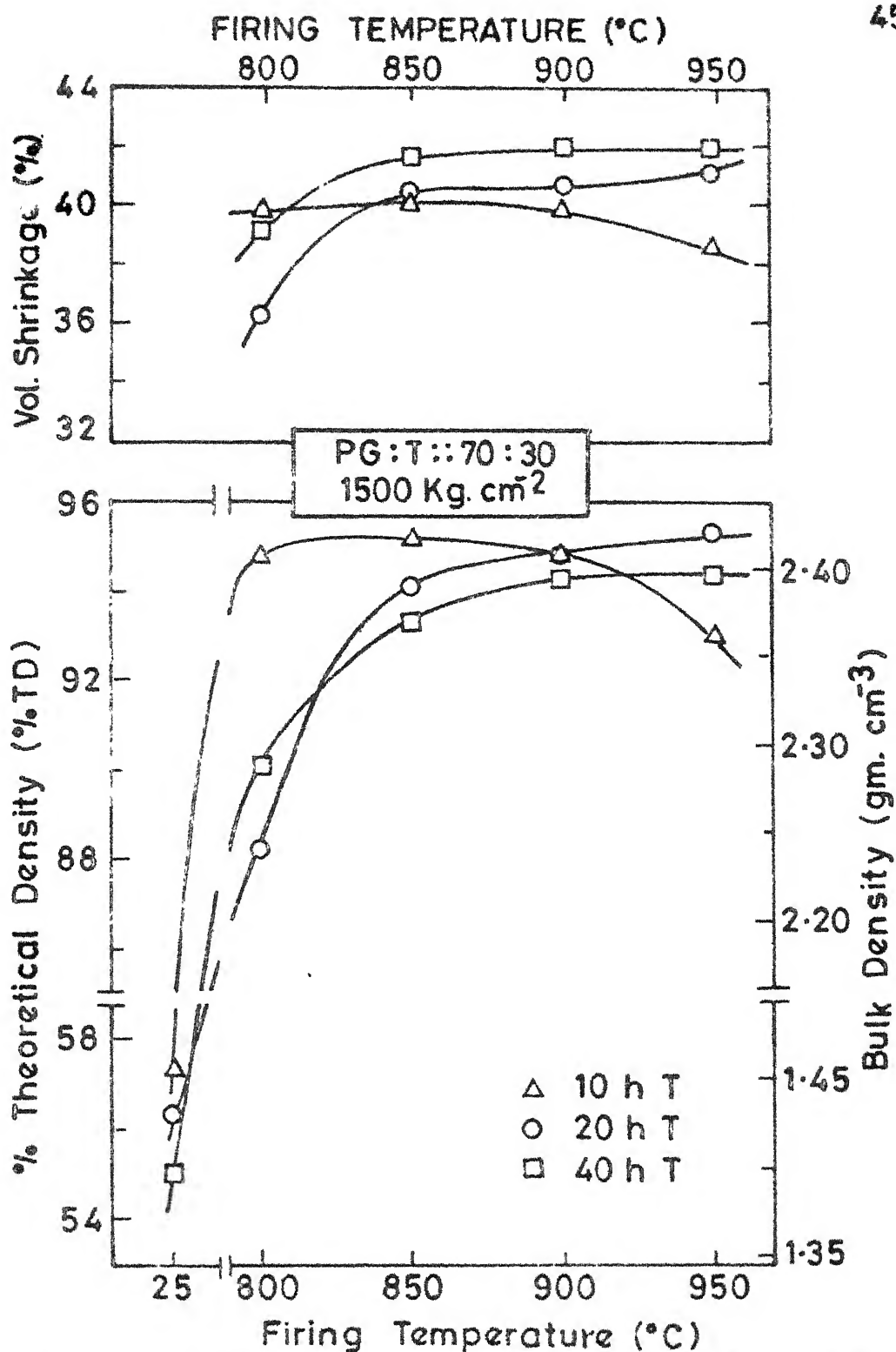


FIG. 5-12. SHRINKAGE, % TD, BULK DENSITY OF PYREX-THERMIT SLAG PELLETS AT DIFFERENT FIRING TEMPERATURES.

as high a densification as given by 40 hours ground powders and not only that, in all cases, the maximum density attained by the coarse powders was at 100 to 150°C lower firing temperatures. It should be however noted that as shown latter, the strength of the bodies were not as high as the bodies with finer fillers.

5.6 Samples for Strength Test

For Quartz, 3 best bodies, and for Alumina and Thermit Slag, 4 best bodies each were selected from the previous set of experiments. For the measurement of the tensile strength by the Brazilian Test, larger samples of 25.4 mm diameter were pressed under 1500 kg cm^{-2} and fired to the same temperature as that of smaller pellets which gave the maximum densification. The fired samples were tested for bulk density by water displacement method, percentage of theoretical density and shrinkage. Water absorption measurements showed that they were mostly in the range of 0.2 to 0.3%, and in no case exceeded 0.5%. The results are shown in tables 5.1, 5.2 and 5.3 for Quartz, Alumina and Thermit Slag respectively.

The strength results for 70% vol bond, are plotted in fig. 5.13. It will be noted that eventhough the percentage of theoretical density (% TD) was almost constant for Alumina bodies and tended to increase some what for the rest, as the fineness of the filler increased, the maximum strength for all three bodies was located at 20 h grinding time. The effect of

filler fineness on strength is only marginal for the Thermit Slag bodies and most pronounced in the Alumina bodies. It is possible to conclude that there is an optimum particle size in order to realize the best properties and not that finer the filler size, better the properties.

Table 5.1 : Data for Quartz bodies

| Code | G | F | E |
|---------------------------------------|---------|---------|---------|
| Quartz, % vol | 30(10h) | 30(20h) | 30(40h) |
| Pyrex glass, % vol | 70 | 70 | 70 |
| Firing Temperature °C | 840 | 840 | 960 |
| Shrinkage, % vol | 40.85 | 42.02 | 43.16 |
| Bulk density, gm cm ⁻³ | 2.247 | 2.253 | 2.290 |
| % Theoretical density | 94.28 | 94.54 | 96.50 |
| Tensile strength, kg cm ⁻² | 149 | 173 | 154 |

Table 5.2 . Data for Alumina bodies

| Code | I | H | A | C |
|--------------------------------------|---------|---------|---------|---------|
| Alumina % vol | 30(10h) | 30(20h) | 30(40h) | 35(40h) |
| Pyrex Glass % vol | 70 | 70 | 70 | 65 |
| Firing Temperature °C | 825 | 930 | 930 | 930 |
| Shrinkage % vol | 38.67 | 41.54 | 41.38 | 41.71 |
| Bulk density gm cm ⁻³ | 2.601 | 2.571 | 2.616 | 2.692 |
| % Theoretical density | 93.87 | 92.67 | 94.46 | 94.23 |
| Tensile strength kg cm ⁻² | 145 | 210 | 145 | 136 |

Table 5.3 : Data for Thermit Slag bodies

| Code | K | J | B | D |
|--------------------------------------|---------|---------|---------|---------|
| Thermit Slag % vol | 30(10h) | 30(20h) | 30(40h) | 35(40h) |
| Pyrex Glass % vol | 70 | 70 | 70 | 65 |
| Firing Temperature °C | 830 | 935 | 935 | 930 |
| Shrinkage % vol | 41.66 | 43.03 | 43.82 | 40.75 |
| Bulk density gm cm ⁻³ | 2.333 | 2.392 | 2.472 | 2.350 |
| % Theoretical density | 91.81 | 94.11 | 97.43 | 90.74 |
| Tensile strength kg cm ⁻² | 89 | 92 | 86 | 105 |

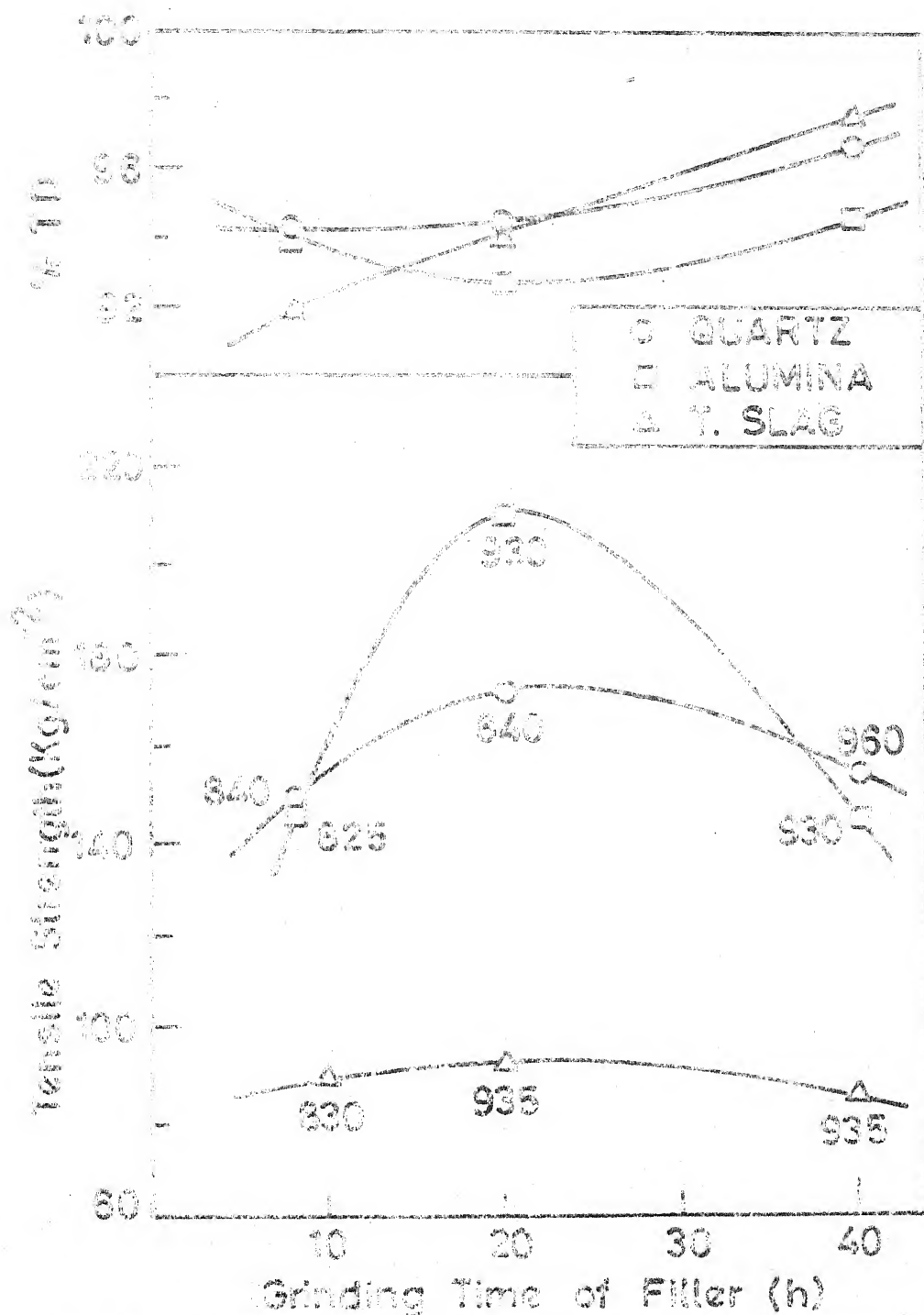



FIG. 5-13. TENSILE STRENGTH AND PERCENTAGE TO AS A FUNCTION OF EXTENT OF GRINDING THE FILLER COMPONENT. FIGURES DENOTE FIRING TEMPERATURE.

CHAPTER-VI

DISCUSSIONS

Of the three glasses tested, Pyrex glass seems to be by far the best for all the three fillers. The reason for its superiority perhaps lies in the fact that its viscosity does not drop with temperature as drastically as in the case of Window glass at least. For E-glass data was not available to permit a similar explanation. This is not to ignore the roll of other factors claimed, for example wetting of filler particles by molten phase, surface tension and its variation with temperature, and possible dissolution of filler, even to a small extent, in the glass. All these factors would differ depending on the nature of the glass.

As shown in figures 5.7 to 5.9, the compaction pressure has an important effect on the ultimate densification reached. This is understandable when one examines the percentage of theoretical densities of green pieces at room temperature. When pressed under 1500 kg cm^{-2} , the green densities tend to go up by approximately 5 to 10% above the densities obtained with 375 kg cm^{-2} pressures. Very roughly, the same increment persists in the densities of the fired samples except for the case of Alumina. In fact Weigmann used isostatic pressing in order to approach 100% theoretical densities. Moreover



there is a definite relationship between the bond content and the moulding pressure. To some extent at least one can be adjusted to compensate for the other as far as the extent of densification is concerned at a given firing temperature.

The firing shrinkages were quite high, of the order of 35 to 40% by volume. This is more or less in agreement with the porosity in the green compact which ranges from 40 to 45% volume. The difference is due to the fact that 100% of theoretical density was not reached. The diametric shrinkages turned out to be 92% to 98% of the shrinkage in height as shown in Appendix III; for pellets of 70% bond content. This could be attributed to the effect of gravity on the pellets which were laid down flat in the furnace. This hypothesis is in confirmity with the fact that when less percentage of bond was used, the two shrinkages tended to be either equal or even their ratio got inverted.

A major outcome of the present investigation seems to be that contrary to the reports in the literature, the fineness of the filler particles must be closely controlled around an optimal size rather than strive for as fine size as possible. If this result can be confirmed by subsequent work, then one must conclude that the cost in grinding the fillers as well as the energy consumed in firing can be reduced still further in these kind of ceramic bodies. This is because, coarser particles reached their maximum densities at lower temperatures than very fine particles.

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CHAPTER-VII

CONCLUSIONS

The following are the principal conclusions arising from this work.

1. Glass bonded ceramic bodies of about 95% of theoretical densities can be made at relatively low firing temperatures of 800-950°C, with Pyrex glass matrix and Alumina, Quartz and Thermit slag fillers.

2. The soak time is about two hours while complete firing cycle required is around 8 to 10 hours, which means a drastic reduction in the firing time as compared to conventional firing practices.

3. For maximum strength, there seems to be an optimal particle size of the filler and not that finer the filler, higher the strength. Moreover coarse particles tend to reach their highest densification at lower temperatures.

4. It is possible to fabricate ceramic bodies having different porosity levels, by decreasing the glassy bond content or firing temperature or moulding pressure for those applications where complete densification is not required.

5. Although three filler materials were studied in this work, it is expected that other ceramic refractory materials like Zircon, Silicon Carbide, etc. can also be bonded in a similar manner.

6. Reproducibility of result is very good.

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APPENDIX-I

CHARACTERISATION OF RAW MATERIALS

A.I.1 True Density (Specific gravity)

True density was determined by pyknometer method.

Powdered material was used and care was taken to remove all the air bubbles from the powders while specific gravity was determined. Water was used as displacement liquid.

Procedure :

| | |
|-------------------------------------|----------------------|
| Volume of specific gravity bottle | = 50 cm ³ |
| Mass of the Bottle | = W gm |
| Mass of (Bottle + Material) | = W ₁ gm |
| Mass of (Bottle + Material + Water) | = W ₂ gm |
| Mass of (Bottle + Water) | = W ₃ gm |

$$\text{True density of material} = \frac{(W_1 - W)(W_3 - W)}{(W_3 - W - W_2 + W_1)50}$$

Table A.I.1 True density (gm cm⁻³) values for raw materials

| Material | Trial No. | | | | Chosen value |
|--------------|-----------|-------|-------|-------|--------------|
| | I | III | III | IV | |
| Window glass | 2.493 | 2.510 | 2.472 | 2.473 | 2.472 |
| E glass | 2.545 | 2.504 | 2.556 | 2.555 | 2.555 |
| Pyrex glass | 2.312 | 2.321 | 2.252 | 2.252 | 2.252 |
| Alumina | 4.006 | 4.016 | 3.973 | 3.981 | 3.930 |
| Quartz | 2.671 | 2.689 | 2.679 | 2.681 | 2.630 |
| Thermit slag | 3.220 | 3.248 | 3.217 | 3.212 | 3.214 |

A.I.2 Fineness of raw materials (Blaine's Specific Surface Area)

The fineness of the raw materials was assessed by their specific surface areas. The latter was determined by using Blaine's apparatus as described in section 4.3.3. All the experiments were conducted at the same room temperature so as to take the viscosity of air constant. Portland cement was used to standardise the apparatus.

Calibration

| | |
|---|---|
| Height of the packed bed | = 1.536 cm |
| Diameter of the packed bed | = 1.25 cms |
| Volume of the packed bed | = 1.855 cm ³ |
| Porosity of the packed bed while standardising (e_s) | = 0.5 |
| Time taken to draw a fixed volume of air to pass through the packed bed while standardising (T_s) | = 31 sec |
| True density of Portland cement (ρ_s) | = 3.15 |
| Specific surface area of Portland cement | = 2250 cm ² gm ⁻¹ |

The formula used for computing the specific surface area is

$$S = S_s \left[\frac{\rho_s (1 - e_s)}{\rho (1 - e)} \right] \cdot \sqrt{\frac{e^3}{e_s^3}} \cdot \sqrt{\frac{T}{T_s}}$$

The various symbols are explained in section 4.3.3.

Table A.I.2 Blaine's Specific surface area values for raw materials

| Material | ρ gm cm^{-3} | e | T sec | S $\text{cm}^2 \text{ gm}^{-1}$ |
|-------------------|-------------------------------|------|----------|------------------------------------|
| Thermit Slag | | | | |
| ground for 40 hrs | 3.214 | 0.60 | 666.56 | 21943 |
| ground for 20 hrs | 3.214 | 0.50 | 647.22 | 21622 |
| ground for 10 hrs | 3.214 | 0.60 | 408.44 | 17177 |
| as received | 3.214 | 0.40 | 69.22 | 4135 |
| Alumina | | | | |
| ground for 40 hrs | 3.980 | 0.55 | 269.22 | 6727 |
| ground for 20 hrs | 3.980 | 0.55 | 127.40 | 4628 |
| ground for 10 hrs | 3.930 | 0.50 | 59.33 | 2464 |
| Quartz | | | | |
| ground for 40 hrs | 2.68 | 0.55 | 321.22 | 9055 |
| ground for 20 hrs | 2.68 | 0.50 | 194.66 | 6626 |
| ground for 10 hrs | 2.68 | 0.50 | 139.45 | 5609 |
| as received | 2.68 | 0.45 | 8.00 | 1042 |
| Window glass | | | | |
| ground for 25 hrs | 2.472 | 0.50 | 249.66 | 8137 |
| Pyrex glass | | | | |
| ground for 25 hrs | 2.252 | 0.50 | 231.44 | 8599 |
| E-glass | | | | |
| ground for 25 hrs | 2.555 | 0.50 | 193.11 | 6923 |

APPENDIX-II

DATA OF PRELIMINARY EXPERIMENTS

Softening temperatures of standard cones made up of Quartz and Window glass were determined. The Full tip, Half bend, Initial tip temperatures of the cones were as follows.

| Material and Vol. Ratio | Tipping temperatures °C | | |
|-------------------------------|-------------------------|-----------|----------|
| | Start | Half bend | Full tip |
| WG; 10 hr Q | | | |
| 75:25 | 708 | 772 | 790 |
| 60:40 | 735 | 816 | 908 |
| 50:50 | 935 | 1250 | 1383 |
| WG; 20 hr Q | | | |
| 75:25 | 660 | 727 | 783 |
| 60:40 | 725 | 863 | 917 |
| 50:50 | 908 | - | - |
| WG; 40 hr Q | | | |
| 75:25 | 730 | 769 | 778 |
| 60:40 | 760 | 881 | 934 |
| 50:50 | 881 | 1150 | - |

APPENDIX-III

EXPERIMENTAL MEASUREMENTS OF BULK DENSITY,
PERCENTAGE THEORETICAL DENSITY AND SHRINKAGE
OF SMALL PELLETS FIRED AT VARIOUS TEMPERATURES

Abbreviations Used

| | | |
|---------|---|---------------------|
| Mat | Material | |
| Vol R | Volume Ratio | |
| Wt R | Weight Ratio | |
| TD | Theoretical Density | |
| Comp Pr | Compaction Pressure used in pellet making | kg cm^{-2} |
| PG | Pyrex glass | |
| EG | E-glass | |
| WG | Window glass | |
| A | Fused Alumina | |
| Q | Quartz | |
| T | Thermit Slag | |
| Q 40 h | Quartz ground for 40 h | |
| FT | Firing temperature | $^{\circ}\text{C}$ |
| H | Height | cm |
| D | Diameter | cm |
| V | Volume | cm^3 |
| BD | Bulk density | gm cm^{-3} |

| Details | FT | % Shrinkage | | | BD | % TD |
|--------------------|------|-------------|-------|-------|-------|-------|
| | | H | D | V | | |
| PELLET No. 31 | 25 | - | - | - | 1.248 | 48.83 |
| Mat =WG; Q 40h | 700 | 7.34 | 7.87 | 21.37 | 1.586 | 62.07 |
| Vol R=60:40 | 750 | 11.92 | 11.62 | 31.22 | 1.811 | 70.88 |
| Wt R =0.580:0.419 | 800 | 9.17 | 9.02 | 24.78 | 1.657 | 64.85 |
| TD =2.555 | 850 | 5.50 | 7.95 | 19.90 | 1.556 | 60.90 |
| Comp Pr = 375 | 900 | 5.11 | 5.35 | 14.93 | 1.469 | 57.50 |
| | 950 | 0.66 | 4.20 | 8.78 | 1.369 | 53.58 |
| | 1000 | -7.21 | 1.38 | -4.29 | 1.192 | 46.65 |
| PELLET No. 32 | 25 | - | - | - | 1.257 | 48.80 |
| Mat = WG; Q 40h | 700 | 2.97 | 2.98 | 8.73 | 1.376 | 53.42 |
| Vol R= 50:50 | 750 | 8.27 | 9.24 | 24.47 | 1.661 | 64.48 |
| Wt R = 0.48:0.52 | 800 | 9.95 | 11.46 | 29.46 | 1.777 | 68.98 |
| TD = 2.576 | 850 | 10.59 | 11.69 | 30.33 | 1.800 | 69.88 |
| Comp Pr = 375 | 900 | 11.63 | 12.91 | 33.01 | 1.871 | 72.63 |
| | 950 | 12.92 | 14.05 | 35.70 | 1.948 | 75.62 |
| | 1000 | 12.01 | 13.98 | 34.93 | 1.923 | 74.65 |
| PELLET No. 33 | 25 | - | - | - | 1.120 | 46.23 |
| Mat = PG; Q 40h | 700 | 4.13 | 4.32 | 12.16 | 1.365 | 56.34 |
| Vol R= 60:40 | 750 | 8.84 | 8.79 | 24.14 | 1.576 | 65.04 |
| Wt R = 0.557:0.443 | 800 | 9.20 | 9.02 | 24.82 | 1.590 | 65.62 |
| TD = 2.423 | 850 | 9.43 | 9.39 | 25.69 | 1.608 | 66.36 |
| Comp Pr = 375 | 900 | 10.14 | 10.08 | 27.32 | 1.643 | 67.81 |
| | 950 | 11.67 | 11.44 | 30.69 | 1.723 | 71.11 |
| | 1000 | 13.76 | 13.64 | 35.51 | 1.843 | 76.06 |
| PELLET No. 34 | 25 | - | - | - | 1.218 | 49.38 |
| Mat = PG; Q 40h | 700 | 1.57 | 1.67 | 4.86 | 1.279 | 51.87 |
| Vol R= 50:50 | 750 | 5.20 | 5.00 | 14.49 | 1.420 | 57.58 |
| Wt R = 0.457:0.543 | 800 | 5.80 | 5.76 | 16.34 | 1.450 | 58.80 |
| TD = 2.466 | 850 | 6.29 | 6.36 | 17.84 | 1.475 | 59.81 |
| Comp Pr = 375 | 900 | 7.38 | 7.12 | 20.14 | 1.516 | 61.48 |
| | 950 | 8.46 | 8.71 | 23.76 | 1.587 | 64.36 |
| | 1000 | 10.12 | 10.68 | 28.36 | 1.688 | 68.45 |

| Details | FT | % Shrinkage | | | BD | % TD |
|--------------------|------|-------------|-------|-------|-------|-------|
| | | H | D | V | | |
| PELLET No. 35 | 25 | - | - | - | 1.276 | 48.97 |
| Mat = EG; Q 40h | 700 | 0.00 | -0.15 | -0.27 | 1.272 | 48.83 |
| Vol R= 60:40 | 750 | 0.50 | -0.08 | 0.37 | 1.275 | 48.94 |
| Wt R = 0.589:0.411 | 800 | 1.87 | 2.05 | 5.86 | 1.347 | 51.71 |
| TD = 2.605 | 850 | 6.37 | 7.06 | 19.12 | 1.566 | 60.12 |
| Comp Pr = 375 | 900 | 9.86 | 9.10 | 25.53 | 1.697 | 65.14 |
| | 950 | 10.11 | 9.56 | 26.53 | 1.719 | 65.99 |
| | 1000 | 10.49 | 9.79 | 27.17 | 1.733 | 66.53 |
| PELLET No. 36 | 25 | - | - | - | 1.269 | 48.47 |
| Mat = EG; Q 40h | 700 | -0.25 | -0.46 | -1.11 | 1.254 | 47.90 |
| Vol R= 50:50 | 750 | -0.13 | -0.30 | -0.74 | 1.257 | 48.01 |
| Wt R = 0.488:0.512 | 800 | 0.63 | 0.76 | 2.12 | 1.293 | 49.39 |
| TD = 2.618 | 850 | 2.89 | 3.11 | 8.85 | 1.389 | 53.03 |
| Comp Pr = 375 | 900 | 5.41 | 5.00 | 14.65 | 1.479 | 56.49 |
| | 950 | 5.41 | 5.16 | 14.93 | 1.481 | 56.57 |
| | 1000 | 5.66 | 5.39 | 15.58 | 1.490 | 56.91 |
| PELLET No. 41 | 25 | - | - | - | 1.519 | 49.37 |
| Mat = WG; A 40h | 700 | 4.18 | 4.06 | 11.95 | 1.653 | 53.73 |
| Vol R= 60:40 | 750 | 9.23 | 8.72 | 24.39 | 1.911 | 62.11 |
| Wt R = 0.482:0.518 | 800 | 11.21 | 10.82 | 29.62 | 2.050 | 66.64 |
| TD = 3.076 | 850 | 12.09 | 11.02 | 30.44 | 2.076 | 67.48 |
| Comp Pr = 375 | 900 | 11.76 | 11.13 | 30.38 | 2.073 | 67.40 |
| | 950 | 11.65 | 11.02 | 30.11 | 2.065 | 67.14 |
| | 1000 | 11.76 | 11.09 | 30.31 | 2.072 | 67.34 |
| PELLET No. 42 | 25 | - | - | - | 1.564 | 48.47 |
| Mat = WG; A 40h | 700 | 0.71 | 0.92 | 2.45 | 1.604 | 49.72 |
| Vol R= 50:50 | 750 | 3.53 | 3.44 | 9.98 | 1.725 | 53.47 |
| Wt R = 0.383:0.617 | 800 | 5.88 | 5.43 | 15.76 | 1.841 | 57.07 |
| TD = 3.226 | 850 | 6.12 | 5.66 | 16.46 | 1.857 | 57.55 |
| Comp Pr = 375 | 900 | 6.24 | 5.58 | 16.39 | 1.856 | 57.53 |
| | 950 | 6.12 | 5.58 | 16.29 | 1.852 | 57.40 |
| | 1000 | 6.35 | 5.62 | 16.64 | 1.858 | 57.59 |

| Details | FT | % Shrinkage | | | BD | % TD |
|--------------------|------|-------------|-------|-------|-------|-------|
| | | H | D | V | | |
| PELLET No. 43 | 25 | - | - | - | 1.459 | 49.57 |
| Mat = PG; A 40h | 700 | 3.52 | 4.09 | 11.29 | 1.644 | 55.86 |
| Vol R= 60:40 | 750 | 8.81 | 8.79 | 24.03 | 1.919 | 65.20 |
| Wt R = 0.459:0.541 | 800 | 11.23 | 11.41 | 30.32 | 2.089 | 71.00 |
| TD = 2.943 | 850 | 13.66 | 13.57 | 35.48 | 2.257 | 76.69 |
| Comp Pr = 375 | 900 | 16.85 | 15.92 | 41.13 | 2.476 | 84.14 |
| | 950 | 18.50 | 17.89 | 45.00 | 2.646 | 89.92 |
| | 1000 | 19.16 | 18.35 | 46.13 | 2.702 | 91.80 |
| PELLET No. 44 | 25 | - | - | - | 1.521 | 48.78 |
| Mat = PG;A40h | 700 | 1.37 | 1.60 | 4.53 | 1.592 | 51.08 |
| Vol R= 50:50 | 750 | 4.11 | 4.33 | 12.25 | 1.729 | 55.47 |
| Wt R = 0.361:0.639 | 800 | 5.94 | 6.08 | 17.11 | 1.827 | 58.60 |
| TD = 3.117 | 850 | 7.08 | 7.37 | 20.30 | 1.900 | 60.99 |
| Comp Pr = 375 | 900 | 8.90 | 9.12 | 24.83 | 2.014 | 64.63 |
| | 950 | 10.96 | 10.94 | 29.36 | 2.144 | 68.78 |
| | 1000 | 12.10 | 11.85 | 31.71 | 2.217 | 71.12 |
| PELLET No. 45 | 25 | - | - | - | 1.566 | 50.14 |
| Mat = EG;A40h | 700 | 0.23 | 0.00 | 0.32 | 1.570 | 50.26 |
| Vol R= 60:40 | 750 | 0.23 | 0.15 | 0.49 | 1.570 | 50.25 |
| Wt R = 0.491:0.509 | 800 | 0.69 | 0.76 | 2.36 | 1.600 | 51.05 |
| TD = 3.124 | 850 | 3.00 | 3.19 | 9.14 | 1.706 | 54.62 |
| Comp Pr = 375 | 900 | 7.85 | 6.99 | 20.33 | 1.946 | 62.68 |
| | 950 | 11.32 | 9.72 | 27.78 | 2.144 | 68.62 |
| PELLET No. 46 | 25 | - | - | - | 1.646 | 50.38 |
| Mat = EG;A40 h | 700 | 0 | 0.23 | 0.53 | 1.653 | 50.61 |
| Vol R= 50:50 | 750 | 0 | 0.30 | 0.53 | 1.654 | 50.63 |
| Wt R = 0.391:0.609 | 800 | 0.24 | 0.33 | 1.06 | 1.654 | 50.62 |
| TD = 3.267 | 850 | 0.96 | 1.06 | 3.17 | 1.683 | 51.53 |
| Comp Pr = 375 | 900 | 3.36 | 2.89 | 8.80 | 1.788 | 54.74 |
| | 950 | 5.03 | 4.40 | 13.20 | 1.878 | 57.49 |

| Details | FT | % Shrinkage | | | BD | % TD |
|--------------------|-----|-------------|-------|-------|-------|-------|
| | | H | D | V | | |
| PELLET No. 51 | 25 | - | - | - | 1.350 | 48.76 |
| Mat = WG; T 40h | 700 | 1.58 | 2.29 | 6.02 | 1.439 | 51.95 |
| Vol R= 60:40 | 750 | 2.70 | 3.28 | 8.86 | 1.469 | 53.04 |
| Wt R = 0.536:0.464 | 800 | 6.30 | 6.34 | 17.72 | 1.612 | 58.22 |
| TD = 2.769 | 850 | 6.76 | 6.56 | 18.56 | 1.610 | 58.20 |
| Comp Pr = 375 | 900 | 6.98 | 6.64 | 18.89 | 1.618 | 58.43 |
| | 950 | 6.98 | 6.64 | 18.89 | 1.616 | 58.36 |
| PELLET No. 53 | 25 | - | - | - | 1.344 | 50.99 |
| Mat = PG; T 40 h | 700 | 1.27 | 1.29 | 3.90 | 1.397 | 53.00 |
| Vol R= 60:40 | 750 | 2.65 | 2.96 | 3.42 | 1.444 | 54.78 |
| Wt R = 0.513:0.487 | 800 | 11.04 | 11.01 | 29.64 | 1.850 | 70.20 |
| TD = 2.636 | 850 | 12.52 | 12.08 | 32.45 | 1.920 | 72.86 |
| Comp Pr = 375 | 900 | 12.74 | 12.23 | 32.76 | 1.930 | 73.21 |
| | 950 | 12.95 | 12.31 | 33.07 | 1.935 | 73.42 |
| PELLET No. 54 | 25 | - | - | - | 1.364 | 49.94 |
| Mat = PG; T 40 h | 700 | 1.32 | 1.29 | 3.88 | 1.420 | 51.95 |
| Vol R= 50:50 | 750 | 1.98 | 2.05 | 5.99 | 1.421 | 51.96 |
| Wt R = 0.412:0.588 | 800 | 5.73 | 5.70 | 16.18 | 1.574 | 57.60 |
| TD = 2.733 | 850 | 9.69 | 9.19 | 25.57 | 1.755 | 64.21 |
| Comp Pr = 375 | 900 | 9.91 | 9.35 | 26.05 | 1.765 | 64.59 |
| | 950 | 9.91 | 9.35 | 26.05 | 1.761 | 64.43 |
| PELLET No. 55 | 25 | - | - | - | 1.502 | 53.28 |
| Mat = EG; T 40 h | 700 | 0.23 | 0.23 | 0.70 | 1.512 | 53.65 |
| Vol R= 60:40 | 750 | 0.47 | 0.46 | 1.38 | 1.509 | 53.53 |
| Wt R = 0.544:0.456 | 800 | 1.41 | 1.75 | 4.84 | 1.545 | 54.82 |
| TD = 2.819 | 850 | 1.88 | 2.89 | 7.60 | 1.577 | 55.92 |
| Comp Pr = 375 | 900 | 4.69 | 5.25 | 14.51 | 1.698 | 60.19 |
| | 950 | 4.93 | 5.34 | 15.03 | 1.706 | 60.53 |
| PELLET No. 52 | 25 | - | - | - | 1.421 | 49.98 |
| Mat = WG; 40 h | 700 | 1.43 | 1.83 | 5.11 | 1.496 | 52.61 |
| Vol R= 50:50 | 750 | 2.14 | 2.38 | 6.70 | 1.506 | 52.98 |
| Wt R = 0.435:0.565 | 800 | 3.33 | 3.36 | 9.70 | 1.535 | 53.98 |
| TD = 2.843 | 850 | 3.80 | 3.74 | 10.93 | 1.543 | 54.26 |
| Comp Pr = 375 | 900 | 3.80 | 3.67 | 10.76 | 1.540 | 54.17 |
| | 950 | 3.80 | 3.82 | 11.11 | 1.545 | 54.35 |

| Details | FT | % Shrinkage | | | BD | % TD |
|--------------------|-----|-------------|-------|-------|-------|-------|
| | | H | D | V | | |
| PELLET No. 56 | 25 | - | - | - | 1.548 | 53.67 |
| Mat = EG; T 40 h | 700 | 0.24 | 0.23 | 0.53 | 1.559 | 54.05 |
| Vol R= 50:50 | 750 | 0.48 | 0.53 | 1.43 | 1.556 | 53.95 |
| Wt R = 0.443:0.557 | 800 | 1.45 | 1.75 | 4.81 | 1.590 | 55.12 |
| TD = 2.884 | 850 | 2.17 | 2.74 | 7.31 | 1.619 | 56.13 |
| Comp Pr = 375 | 900 | 3.62 | 3.96 | 11.05 | 1.677 | 58.15 |
| | 950 | 3.62 | 3.81 | 10.70 | 1.664 | 57.69 |
| PELLET No. 71 | 25 | - | - | - | 1.450 | 49.31 |
| Mat = PG; A 40h | 750 | 8.64 | 8.51 | 23.52 | 1.896 | 64.42 |
| Vol R= 60:40 | 800 | 11.88 | 11.47 | 30.84 | 2.092 | 71.10 |
| Wt R = 0.459:0.541 | 850 | 14.04 | 13.60 | 35.77 | 2.252 | 76.52 |
| TD = 2.943 | 900 | 16.20 | 15.35 | 39.90 | 2.404 | 81.67 |
| Comp Pr = 375 | 950 | 18.36 | 17.25 | 44.04 | 2.585 | 87.82 |
| PELLET No. 72 | 25 | - | - | - | 1.619 | 55.06 |
| Mat = PG; A 40h | 750 | 7.31 | 7.03 | 19.89 | 2.023 | 68.74 |
| Vol R= 60:40 | 800 | 10.24 | 9.76 | 27.00 | 2.213 | 75.10 |
| Wt R = 0.459:0.541 | 850 | 12.44 | 11.50 | 31.44 | 2.353 | 79.93 |
| TD = 2.943 | 900 | 14.15 | 13.01 | 35.17 | 2.487 | 84.50 |
| Comp Pr = 1500 | 950 | 16.10 | 14.52 | 38.72 | 2.632 | 89.43 |
| PELLET No. 73 | 25 | - | - | - | 1.396 | 49.79 |
| Mat = PG; A 40h | 750 | 10.06 | 10.17 | 27.36 | 1.960 | 68.61 |
| Vol R= 65:35 | 800 | 13.70 | 13.59 | 35.53 | 2.205 | 77.19 |
| Wt R = 0.512:0.488 | 850 | 15.85 | 15.49 | 39.94 | 2.359 | 82.57 |
| TD = 2.857 | 900 | 17.56 | 17.16 | 43.40 | 2.506 | 87.72 |
| Comp Pr = 375 | 950 | 19.06 | 18.38 | 46.07 | 2.629 | 92.02 |
| PELLET No. 74 | 25 | - | - | - | 1.557 | 54.49 |
| Mat = PG; A 40h | 750 | 9.93 | 9.66 | 26.68 | 2.114 | 73.99 |
| Vol R= 65:35 | 800 | 13.32 | 12.45 | 33.55 | 2.331 | 81.59 |
| Wt R = 0.512:0.488 | 850 | 15.35 | 14.26 | 37.81 | 2.489 | 87.12 |
| TD = 2.859 | 900 | 16.70 | 15.40 | 40.43 | 2.598 | 90.91 |
| Comp Pr = 1500 | 950 | 17.16 | 15.92 | 41.41 | 2.645 | 92.57 |

| Details | FT | % Shrinkage | | | BD | % TD |
|--------------------|-----|-------------|-------|-------|-------|-------|
| | | H | D | V | | |
| PELLET No. 75 | 25 | - | - | - | 1.357 | 48.99 |
| Mat = PG; A 40h | 750 | 13.70 | 13.59 | 35.59 | 2.106 | 76.03 |
| Vol R= 70:30 | 800 | 17.38 | 16.92 | 43.09 | 2.380 | 85.95 |
| Wt R = 0.569:0.431 | 850 | 19.22 | 18.45 | 46.40 | 2.524 | 91.11 |
| TD = 2.770 | 900 | 19.94 | 18.98 | 47.45 | 2.577 | 93.02 |
| Comp Pr = 375 | 950 | 19.84 | 18.98 | 47.30 | 2.573 | 92.90 |
| PELLET No. 76 | 25 | - | - | - | 1.499 | 54.13 |
| Mat = PG; A 40h | 750 | 11.67 | 11.31 | 30.46 | 2.158 | 77.91 |
| Vol R= 70:30 | 800 | 15.64 | 14.71 | 38.56 | 2.430 | 87.73 |
| Wt R = 0.569:0.431 | 850 | 17.18 | 15.76 | 41.31 | 2.537 | 91.61 |
| ID = 2.770 | 900 | 17.62 | 16.37 | 42.42 | 2.538 | 93.43 |
| Comp Pr = 1500 | 950 | 17.62 | 16.29 | 42.26 | 2.583 | 93.27 |
| PELLET No. 77 | 25 | - | - | - | 1.417 | 54.71 |
| Mat = PG; T 40h | 750 | 5.91 | 5.97 | 16.66 | 1.703 | 65.76 |
| Vol R= 65:35 | 800 | 13.95 | 12.84 | 34.54 | 2.154 | 83.19 |
| Wt R = 0.565:0.435 | 850 | 15.13 | 13.82 | 36.94 | 2.231 | 86.18 |
| TD = 2.589 | 900 | 15.13 | 13.97 | 37.29 | 2.245 | 86.70 |
| Comp Pr = 1500 | 950 | 15.37 | 14.12 | 37.63 | 2.259 | 87.26 |
| PELLET No. 78 | 25 | - | - | - | 1.398 | 55.01 |
| Mat = PG; T 40h | 750 | 7.72 | 7.92 | 21.73 | 1.787 | 70.32 |
| Vol R= 70:30 | 800 | 15.69 | 15.09 | 39.22 | 2.289 | 90.07 |
| W5 R = 0.62:0.38 | 850 | 17.10 | 16.00 | 41.60 | 2.372 | 93.35 |
| TD = 2.541 | 900 | 17.33 | 16.23 | 41.94 | 2.395 | 94.24 |
| Comp Pr = 1500 | 950 | 17.56 | 16.06 | 41.94 | 2.396 | 94.28 |
| PELLET No. 81 | 25 | - | - | - | 1.348 | 47.66 |
| Mat = PG; T 40h | 750 | 1.89 | 2.13 | 6.09 | 1.435 | 50.71 |
| Vol R= 40:60 | 800 | 4.49 | 4.71 | 13.22 | 1.543 | 54.53 |
| Wt R = 0.318:0.682 | 850 | 6.38 | 6.31 | 17.74 | 1.641 | 57.03 |
| TD = 2.83 | 900 | 6.62 | 6.38 | 18.08 | 1.625 | 57.40 |
| Comp Pr = 375 | 950 | 6.62 | 6.46 | 18.26 | 1.620 | 57.27 |

| Details | FT | % Shrinkage | | | BD | % TD |
|--------------------|------|-------------|-------|-------|-------|-------|
| | | H | D | V | | |
| PELLET No. 82 | 25 | - | - | - | 1.479 | 56.09 |
| Mat = PG; T 40h | 750 | 3.91 | 4.61 | 12.66 | 1.691 | 64.15 |
| Vol R= 60:40 | 800 | 11.25 | 9.91 | 27.99 | 2.040 | 77.39 |
| Wt R = 0.512:0.488 | 850 | 11.98 | 10.59 | 29.59 | 2.086 | 79.13 |
| TD = 2.636 | 900 | 11.98 | 10.74 | 29.76 | 2.093 | 79.40 |
| Comp Pr = 1500 | 950 | 11.98 | 10.82 | 29.95 | 2.091 | 79.34 |
| PELLET No. 83 | 25 | - | - | - | 1.333 | 55.01 |
| Mat = PG; Q 40h | 750 | 6.08 | 6.70 | 18.25 | 1.630 | 67.28 |
| Vol R= 60:40 | 800 | 7.84 | 9.33 | 24.19 | 1.757 | 72.53 |
| Wt R = 0.588:0.412 | 850 | 10.00 | 9.78 | 26.73 | 1.818 | 75.02 |
| TD = 2.423 | 900 | 10.78 | 10.38 | 28.29 | 1.858 | 76.70 |
| Comp Pr = 1500 | 950 | 12.16 | 11.59 | 31.26 | 1.939 | 80.03 |
| | 1000 | 13.72 | 13.54 | 35.50 | 2.065 | 85.21 |
| PELLET No. 84 | 25 | - | - | - | 1.299 | 54.09 |
| Mat = PG; Q 40 h | 750 | 10.45 | 11.10 | 29.23 | 1.836 | 76.44 |
| Vol R= 65:35 | 800 | 11.64 | 11.93 | 31.52 | 1.892 | 78.76 |
| Wt R = 0.609:0.391 | 850 | 11.64 | 12.01 | 31.52 | 1.895 | 78.90 |
| TD = 2.402 | 900 | 11.83 | 12.16 | 31.95 | 1.904 | 79.26 |
| Comp Pr = 1500 | 950 | 12.43 | 12.76 | 33.38 | 1.941 | 80.81 |
| | 1000 | 12.62 | 13.74 | 34.95 | 1.990 | 82.85 |
| PELLET No. 85 | 25 | - | - | - | 1.289 | 54.07 |
| Mat = PG; Q 40 h | 750 | 13.88 | 14.34 | 36.79 | 2.039 | 85.57 |
| Vol R= 70:30 | 800 | 14.69 | 14.87 | 38.10 | 2.079 | 87.25 |
| Wt R = 0.662:0.337 | 850 | 14.69 | 15.02 | 38.39 | 2.037 | 87.56 |
| TD = 2.383 | 900 | 15.09 | 15.17 | 38.83 | 2.102 | 88.19 |
| Comp Pr = 1500 | 950 | 15.49 | 15.55 | 39.71 | 2.130 | 89.40 |
| | 1000 | 15.29 | 15.62 | 39.71 | 2.130 | 89.40 |
| PELLET No. 86 | 25 | - | - | - | 1.455 | 57.28 |
| Mat = PG; T 10h | 750 | 11.75 | 12.79 | 32.87 | 2.163 | 85.33 |
| Vol R= 70:30 | 800 | 15.57 | 15.52 | 39.84 | 2.409 | 94.78 |
| Wt R = 0.62:0.38 | 850 | 15.85 | 15.67 | 40.24 | 2.418 | 95.17 |
| TD = 2.541 | 900 | 15.57 | 15.39 | 39.84 | 2.410 | 94.83 |
| Comp Pr = 1500 | 950 | 14.75 | 15.14 | 38.65 | 2.361 | 92.91 |

| Details | FT | % Shrinkage | | | BD | % TD |
|--------------------|------|-------------|-------|-------|-------|-------|
| | | H | D | V | | |
| PELLET No. 87 | 25 | - | - | - | 1.609 | 58.09 |
| Mat = PG; A 10h | 800 | 15.63 | 14.49 | 38.22 | 2.608 | 94.15 |
| Wt R = 70:30 | 850 | 15.87 | 14.49 | 38.39 | 2.616 | 94.43 |
| Wt R = 0.569:0.431 | 900 | 15.38 | 14.34 | 37.87 | 2.586 | 93.36 |
| TD = 2.770 | 950 | 14.18 | 13.96 | 36.47 | 2.528 | 91.25 |
| Comp Pr = 1500 | | | | | | |
| PELLET No. 88 | 25 | - | - | - | 1.425 | 59.86 |
| Mat = PG; Q 10h | 800 | 15.21 | 14.76 | 38.35 | 2.159 | 90.59 |
| Vol R= 70:30 | 850 | 15.21 | 14.76 | 38.35 | 2.161 | 90.67 |
| Wt R = 0.662:0.337 | 900 | 15.63 | 14.91 | 38.95 | 2.179 | 91.44 |
| TD = 2.383 | 950 | 15.63 | 15.14 | 39.25 | 2.191 | 91.92 |
| Comp Pr = 1500 | 1000 | 14.58 | 14.91 | 38.20 | 2.155 | 90.04 |
| PELLET No. 89 | 25 | - | - | - | 1.430 | 56.29 |
| Mat = PG; T 20h | 800 | 14.19 | 13.77 | 36.33 | 2.241 | 88.20 |
| Vol R= 70:30 | 850 | 16.70 | 15.36 | 40.33 | 2.396 | 94.11 |
| Wt R = 0.620:0.380 | 900 | 16.93 | 15.50 | 40.66 | 2.406 | 94.70 |
| TD = 2.541 | 950 | 17.16 | 15.73 | 41.16 | 2.423 | 95.37 |
| Comp Pr = 1500 | | | | | | |
| PELLET No. 90 | 25 | - | - | - | 1.592 | 57.46 |
| Mat = PG; A 20h | 750 | 11.37 | 14.54 | 35.27 | 2.459 | 88.79 |
| Vol R= 70:30 | 800 | 14.69 | 13.79 | 36.64 | 2.510 | 90.63 |
| Wt R = 0.569:0.431 | 850 | 15.64 | 14.62 | 38.53 | 2.588 | 93.44 |
| TD = 2.770 | 900 | 16.11 | 14.85 | 39.21 | 2.617 | 94.47 |
| Comp Pr = 1500 | 950 | 16.11 | 14.85 | 39.21 | 2.617 | 94.47 |
| PELLET No. 91 | 25 | - | - | - | 1.369 | 57.44 |
| Mat = PG; Q 20h | 750 | 10.23 | 10.77 | 28.57 | 1.915 | 80.38 |
| Vol R= 70:30 | 800 | 13.18 | 13.34 | 34.81 | 2.094 | 87.89 |
| Wt R = 0.662:0.337 | 850 | 13.41 | 13.49 | 35.30 | 2.107 | 88.43 |
| TD = 2.383 | 900 | 13.64 | 13.64 | 35.63 | 2.120 | 88.96 |
| Comp Pr = 1500 | 950 | 14.09 | 14.02 | 36.45 | 2.150 | 90.22 |
| | 1000 | 13.41 | 13.94 | 35.96 | 2.127 | 89.25 |

APPENDIX-IV

EXPERIMENTAL MEASUREMENTS OF BULK DENSITY, PERCENTAGE THEORETICAL DENSITY, SHRINKAGE, WATER ABSORPTION AND TENSILE STRENGTH OF BIG SAMPLES

Abbreviations used other than those in Appendix III

T.S Tensile Strength

* Reading not considered

± Volume of the standard samples was found
by water displacement method

Water Ab Water Absorption

| Details | No | % Shrinkage | | | BD | % TD | %Water Ab | T.S |
|--------------------|----|-------------|-------|-------|-------|-------|--------------|------|
| | | H | D | V+ | | | | |
| CODE A | | | | | | | | |
| Mat = PG;A 40h | A1 | 16.88 | 16.03 | 41.42 | 2.605 | 94.06 | 0.272 | |
| Vol R= 70:30 | A2 | 16.87 | 16.22 | 41.02 | 2.616 | 94.45 | 0.276 | |
| Wt R = 0.569:0.431 | A3 | 17.26 | 16.22 | 41.23 | 2.637 | 95.21 | 0.191 | 134* |
| TD = 2.770 | A4 | 17.18 | 15.99 | 41.39 | 2.606 | 94.11 | 0.320 | |
| Comp Pr = 1500 | A5 | 16.84 | 15.87 | 41.29 | 2.635 | 94.05 | 0.346 | |
| F.T = 930 | A6 | 17.81 | 15.99 | 41.93 | 2.627 | 94.85 | 0.255 | 145 |
| Average Values | | 17.14 | 16.05 | 41.38 | 2.616 | 94.46 | 0.277 | 145 |
| CODE B | | | | | | | | |
| Mat = PG;T 40h | B1 | 17.32 | 17.09 | 43.37 | 2.498 | 98.30 | 0.224 | |
| Vol R= 70:30 | B2 | 17.51 | 17.44 | 43.76 | 2.497 | 98.26 | 0.279 | |
| Wt R = 0.62:0.33 | B3 | 18.16 | 17.40 | 44.27 | 2.470 | 97.20 | 0.366 | 86 |
| TD = 2.541 | B4 | 18.09 | 17.37 | 43.91 | 2.458 | 96.74 | 0.302 | 79* |
| Comp Pr = 1500 | B5 | 17.96 | 17.33 | 43.81 | 2.457 | 96.71 | 0.374 | |
| F.T = 935 | B6 | - | - | - | 2.463 | 96.93 | 0.284 | |
| Average Values | | 17.84 | 17.33 | 43.82 | 2.474 | 97.43 | 0.288 | 86 |
| CODE C | | | | | | | | |
| Mat = PG; A 40h | C1 | 17.05 | 15.83 | 40.81 | 2.590 | 94.16 | 0.191 | 136 |
| Vol R= 65:35 | C2 | 17.34 | 16.06 | 42.22 | 2.683 | 93.90 | 0.276 | 110* |
| Wt R = 0.512:0.488 | C3 | 16.84 | 16.18 | 42.39 | 2.703 | 94.62 | 0.249 | |
| TD = 2.857 | | | | | | | | |
| Comp Pr = 1500 | | | | | | | | |
| F.T = 930 | | | | | | | | |
| Average Values | | 17.74 | 16.02 | 41.71 | 2.692 | 94.23 | 0.239 | 136 |
| CODE D | | | | | | | | |
| Mat = PG;T 40h | D1 | 16.96 | 15.53 | 41.12 | 2.353 | 90.87 | 0.356 | |
| Vol R= 65:35 | D2 | 16.68 | 15.37 | 40.18 | 2.327 | 89.87 | 0.293 | |
| Wt R = 0.565:0.435 | D3 | 16.45 | 15.25 | 40.28 | 2.351 | 90.82 | 0.307 | |
| TD = 2.589 | D4 | 16.54 | 15.29 | 40.35 | 2.340 | 90.39 | 0.285 | |
| Comp Pr = 1500 | D5 | 16.56 | 15.25 | 40.54 | 2.354 | 90.92 | 0.197 | 105 |
| F.T = 930 | D6 | 17.12 | 15.72 | 41.37 | 2.352 | 90.84 | 0.312 | 71* |
| | D7 | 17.25 | 15.64 | 41.39 | 2.369 | 91.49 | 0.193 | |
| Average Values | | 16.79 | 15.44 | 40.75 | 2.350 | 90.74 | 0.278 | 105 |

| Details | No | % Shrinkage | | | BD | %TD | Percent water Ab. | T. |
|--------------------|----|-------------|-------|----------------|--------|--------|-------------------------|----|
| | | H | D | V ⁺ | | | | |
| CODE E | E1 | 17.62 | 16.72 | 44.13 | 2.295 | 96.32 | 0.292 | |
| Mat = PG; Q 40h | E2 | 12.98* | 16.37 | 42.69 | 2.276 | 96.53 | 0.344 | |
| Vol R= 70:30 | E3 | 17.28 | 16.49 | 42.63 | 2.287 | 95.96 | 0.322 | 10 |
| Wt R = 0.662:0.337 | E4 | 17.35 | 16.87 | 42.83 | 2.288 | 96.01 | 0.189 | 15 |
| TD = 2.383 | E5 | 17.68 | 16.49 | 43.52 | 2.305 | 96.72 | 0.319 | |
| Comp Pr = 1500 | | | | | | | | |
| E.T = 960 | | | | | | | | |
| Average values | | 17.48 | 16.59 | 43.16 | 2.290 | 96.50 | 0.293 | 15 |
| CODE F | | | | | | | | |
| Mat = PG; Q 20 h | F1 | 16.74 | 16.17 | 41.54 | 2.241 | 94.06 | 0.549 | |
| Vol R= 70:30 | F2 | 17.25 | 16.40 | 42.63 | 2.275 | 95.49 | 0.306 | 17 |
| Wt R = 0.662:0.337 | F3 | 16.65 | 15.90 | 41.43 | 2.233 | 93.70 | 0.444 | 11 |
| TD = 2.383 | F5 | 17.30 | 16.52 | 42.46 | 2.276 | 95.52 | 0.440 | |
| Comp Pr = 1500 | F6 | 17.16 | 16.29 | 42.05 | 2.339 | 93.95 | 0.436 | |
| F.T.= 840 | | | | | | | | |
| Average values | | 17.02 | 16.26 | 42.02 | 2.253 | 94.54 | 0.435 | 17 |
| CODE G | | | | | | | | |
| Mat = PG; Q 10h | G1 | 17.16 | 15.78 | 41.85 | 2.248 | 94.33 | 0.364 | |
| Vol R= 70:30 | G2 | 15.76 | 15.51 | 40.76 | 2.241 | 94.04 | 0.347 | |
| Wt R = 0.662:0.337 | G3 | 16.20 | 15.74 | 35.21 | 2.052* | 86.12* | 0.336 | 14 |
| TD = 2.383 | G4 | 16.40 | 15.70 | 40.79 | 2.246 | 94.24 | 0.644 | 11 |
| Comp Pr = 1500 | G5 | 16.26 | 15.70 | 40.87 | 2.259 | 94.80 | 0.351 | |
| F.T = 840 | | | | | | | | |
| Average Values | | 16.36 | 15.69 | 40.85 | 2.247 | 94.28 | 0.408 | 14 |
| CODE H | | | | | | | | |
| Mat = PG; A 20h | H1 | 16.08 | 15.78 | 40.46 | 2.555 | 92.25 | 0.52 | 21 |
| Vol R= 70:30 | H2 | 22.51* | 15.62 | 45.08 | 2.579 | 93.12 | 0.35 | |
| Wt R = 0.569:0.431 | H3 | 18.94 | 15.23 | 41.90 | 2.552 | 92.14 | 0.36 | |
| TD = 2.770 | H4 | 13.24 | 15.93 | 38.70 | 2.571 | 92.82 | 0.31 | |
| Comp Pr = 1500 | H5 | 9.6* | 15.47 | 35.72* | 2.576 | 93.01 | 0.27 | 10 |
| F.T = 930 | | | | | | | | |
| Average values | | 16.09 | 15.61 | 41.54 | 2.571 | 92.67 | 0.36 | 21 |

| Details | No | % Shrinkage | | | BD | %TD | Percent water Ab | T. |
|--------------------|----|-------------|--------|----------------|-------|-------|------------------------|----|
| | | H | D | V ^r | | | | |
| CODE I | | | | | | | | |
| Mat = PG; A 10h | I1 | 15.13 | 14.60 | - | 2.598 | 93.80 | 0.37 | |
| Vol R= 70:30 | I2 | 14.96 | 14.44 | 38.66 | 2.603 | 93.98 | 0.28 | 14 |
| Wt R = 0.569:0.431 | I3 | 15.38 | 14.68 | 38.38 | 2.589 | 93.48 | 0.27 | |
| TD = 2.770 | I4 | 15.44 | 14.87 | 38.96 | 2.619 | 94.55 | 0.29 | 14 |
| Comp Pr = 1500 | I5 | 11.41* | 13.21* | 35.24* | 2.591 | 93.53 | 0.34 | |
| F.T. = 825 | | | | | | | | |
| Average values | | 15.24 | 14.65 | 38.67 | 2.601 | 93.87 | 0.31 | 14 |
| CODE J | | | | | | | | |
| Mat = PG; T 20 h | J1 | 17.86 | 16.48 | 42.42 | 2.388 | 93.96 | 0.37 | |
| Vol R= 70:30 | J2 | 20.95* | 16.97 | 44.84 | 2.389 | 94.05 | 0.32 | |
| Wt R = 0.62:0.38 | J3 | 17.86 | 16.52 | 42.59 | 2.396 | 94.28 | 0.31 | |
| TD = 2.541 | J4 | 18.12 | 16.48 | 42.68 | 2.395 | 94.25 | 0.34 | 9 |
| Comp Pr = 1500 | J5 | 17.10 | 16.40 | 42.61 | 2.394 | 94.23 | 0.38 | |
| F.T. = 935 | | | | | | | | |
| Average values | | 17.73 | 16.57 | 43.03 | 2.392 | 94.11 | 0.34 | 92 |
| CODE K | | | | | | | | |
| Mat = PG; T 10 h | K1 | 17.60 | 16.08 | 41.78 | 2.351 | 92.52 | 0.33 | |
| Vol R= 70:30 | K2 | 12.94* | 15.31 | 41.43 | 2.332 | 91.79 | 0.41 | 29 |
| Wt R = 0.62:0.38 | K3 | 17.67 | 16.28 | 42.32 | 2.338 | 92.02 | 0.35 | 52 |
| TD = 2.541 | K4 | 17.64 | 15.81 | 41.44 | 2.312 | 90.99 | 0.73 | |
| Comp Pr = 1500 | K5 | 17.39 | 15.81 | 41.35 | 2.331 | 91.73 | 0.45 | |
| F.T. = 830 | | | | | | | | |
| Average values | | 17.58 | 15.98 | 41.66 | 2.333 | 91.81 | 0.45 | 89 |